VISIT…

Chapter 3

Lubricant Testing*

Virtually every lubricant or other petroleum product is manufactured to certain performance standards or specifications. It will exhibit certain properties or characteristics which the manufacturer describes in his sales literature, data sheets, or related documents. These descriptions may range from somewhat superficial in common household products to highly technical and sophisticated in specialty products for industry.

Whenever the industrial user is involved in the selection, or faced with the optimized application of petroleum products, he will find himself confronted by terms and descriptions that relate to these product properties and characteristics. Sometimes called "typical inspections," the properties of just one multi-purpose grease include such items as *worked penetration, dropping point, viscosity, oil separation, wheel bearing leakage, Timken OK load, four-ball wear test, water washout*, and *corrosion prevention rating*. The question is, what do these terms mean, and how important are they?

Since the scope and intent of our text is aimed at conveying practical knowledge to the reader, we must enable him or her to make comparisons among products. With this in mind, we have endeavored to describe the most important tests and their significance to the lubricant user. We deliberately opted to leave out many of the detailed descriptions of test apparatus and testing procedures of interest to the laboratory technician, but did include them in the few instances where clarity called for more detail.

AIR ENTRAINMENT

DIN 51 381 TUV Impinger Test

Air entrained in a lubricating oil can disrupt the lubricating film and cause excessive wear of the surfaces involved. In hydraulic systems, because entrained air is compressible, it can cause erratic and inefficient operation of the system.

The term "air entrainment" refers to a dispersion of air bubbles in oil in which the bubbles are so small that they tend to rise very slowly to the air-oil interface. The presence of the bubbles gives the oil a hazy or cloudy appearance.

There is no standard method for testing the air entrainment characteristics of oil. The American Society for Testing Materials is in the process of investigating various tests

*Source: Exxon Company U.S.A., Houston, Texas.

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with the idea of standardizing on one. The German DIN 51 381 "TUV Impinger Test" is primarily used to test steam turbine oils and hydraulic fluids for their air release properties. It is the standard generally accepted in Europe and is being considered as the ASTM method.

Significance

Air entrainment consists of slow-rising bubbles dispersed throughout an oil and is to be distinguished from foaming, which consists of bubbles that rise quickly to the surface of the oil. Both of these conditions are undesirable in a lubricating system. However, it is often difficult to distinguish between them because of high flow rates and turbulence in the system. Relatively small amounts of air are involved in air entrainment while larger amounts are involved with foaming. These conditions are considered separate phenomena and are measured in separate laboratory tests. Entrained air is not a normal condition; it is primarily caused by mechanical problems. Some of these are:

Insufficient Reservoir Fluid Level: Air can be drawn into the pump suction along with the oil.

Systems Leaks: Air can be introduced into the oil at any point in the system where the pressure is below atmospheric pressure.

Improper Oil Addition Methods: If make-up oil is added in a manner that causes splashing, it is possible for air to become entrained in the oil.

Faulty System Design: Design faults involve such things as placement of oil return so that the returning oil splashes into the reservoir, or placement of the pump next to the return opening.

The current trend in hydraulic oil systems, turbine oil systems, and industrial circulating oil systems of every kind is to decrease reservoir size and increase flow rates and system pressures. This trend increases the tendency for air entrainment, thereby making the air release property of an oil more significant.

Some additives used to reduce foaming tend to increase the air entrainment tendency of an oil. The choice of an anti-foam additive requires striking a balance between these two undesirable phenomena.

ANILINE POINT

ASTM D 611 and ASTM D 1012

Many petroleum products, particularly the lighter ones, are effective solvents for a variety of other substances. The degree of solvent power of the petroleum product varies with the types of hydrocarbons included in it. Frequently it is desirable to know what this solvent power is, either as a favorable characteristic in process applications where good solvency is important, or as an unfavorable characteristic when the product may contact materials susceptible to its solvent action.

The aniline point determination is a simple test, easily performed in readily available equipment. In effect, it measures the solvent power of the petroleum product for aniline, an aromatic substance. The solvent powers for many other materials are related

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to the solvent power for aniline.

Aniline is at least partially soluble in almost all hydrocarbons, and its degree of solubility in any particular hydrocarbon increases as the temperature of the mixture is increased. When the temperature of complete solubility is reached, the mixture is a clear solution; at lower temperatures, the mixture is turbid. The test procedures make use of this characteristic by measuring the temperature at which the mixture clouds as it is cooled. The greater the solvent power of the hydrocarbon for aniline, the lower the temperature at which cloudiness first appears.

Usually, paraffinic hydrocarbons have the least solvency for aniline (and most other materials) and consequently have the highest aniline points. Aromatics have the greatest solvency and the lowest aniline points (usually well below room temperature), while naphthenic materials are intermediate between the paraffins and the aromatics.

Significance of Results

Aniline point is most significant for solvents, since it is one indication of solvent power. In general, the lower the aniline point of a product, the greater its solvent power. Other available laboratory tests measure the solvent power of the product for the specific type of substance with which it will be used. Two tests of this type, which have been standardized and accepted, are used to determine kauri-butanol value and nitro-cellulose diluting power. However, these latter tests are much more complicated than the aniline point determination, and small laboratories do not usually have the facilities for performing them. The choice of tests is usually dictated by which one correlates best in a particular application.

Aniline point is useful in predicting the ignition characteristics of diesel fuels. For this purpose, the aniline point is used in conjunction with the API gravity of the fuel to determine its "diesel index." This procedure is described later, "Cetane Number," since diesel index and cetane number are used for similar purposes. Diesel index, in turn, is useful in estimating the enrichment value of oils used for gas enrichment.

For a lubricating or hydraulic oil, aniline point is an indication of its tendency to cause softening and swelling of rubber parts contacted by the oil. The lower the aniline point, the greater the swelling tendency. Aniline point is also used as a factor in determining the relative compatibility of a rubber plasticizer with a rubber formulation.

ASH CONTENT

ASTM D 874 for Sulfated Residue

The ash content of a lubricating oil is related to the quantity of incombustible materials that may be present. Though a straight distillate mineral oil is, in itself, nearly ashless, certain lubricants are formulated with solutions of metallic additives that will not be completely burned. The ash left by these products may be appreciable. Used oils, moreover, may be contaminated with dirt and abraded metals that likewise appear as ash after the oil itself has been consumed.

The percentage of ash that remains after oil has been burned, therefore, gives an

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indication of the quantity of metallic additive, non-combustible solid contamination, or both, that the oil may contain. Significance of ash content depends on the type of oil, its condition, and the actual test by which it is evaluated.

The simplest method of determining the ash content of a lubricating oil is to burn a sample of known weight, applying sufficient heat to consume all of the combustibles. The weight of the residue that is left establishes a value for determining the percentage of ash. This procedure is described under the ASTM designation D 482.

In general, however, the preferred test is that specified under the ASTM method D 874 for "sulfated residue." Here, the oil is first strained to remove solid contaminants; then it is burned under controlled conditions. After the burning, the residue is treated with sulfuric acid to assure consistent degrees of oxidation of all components. Acid treatment improves the uniformity of the results, making them more reliable.

There is still another method, ASTM D 810, which also yields sulfated residue, but which serves primarily to determine the percentages of lead, iron, or copper. With lubricating oils, this sort of analysis is generally of lesser significance.

Significance of Results

Many oils for internal combustion engines are formulated with detergent additives based on metallic derivatives such as those of barium or calcium. These additives help to keep the engine clean. Being metallic, these materials appear in one chemical form or another in the ash.

For new oils of this type, therefore, sulfated ash may serve as a manufacturer's check on proper formulation. An abnormal ash may indicate a change in additive content and, hence, a departure from an established formulation.

For new oils of unknown formulation, sulfated ash is sometimes accepted as a rough indication of detergent level. The principle is based on the dubious assumption that a higher percent of ash implies a stronger concentration of detergent and, hence, an oil of greater cleanliness properties. As a means of evaluating detergency, however, the test for ash is far less reliable than the usual engine and field tests, the primary advantage of ash content lying in the expediency with which it is determined.

There are several reasons why the relationship between sulfated residue and detergency may be extremely distorted:

1. Detergency depends on the properties of the base oil as well as on the additive. Some combinations of base oil and additive are much more effective than others.

2. Detergents vary considerably in their potency, and some leave more ash than others. Detergents have been developed, in fact, that leave no ash at all.

3. Some of the ash may be contributed by additives other than detergents.

4. There appears to be a limit to the effective concentration of detergent. Nothing is gained by exceeding this limit, and a superabundance of detergent may actually reduce cleanliness.

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Sulfated ash has also been used to determine additive depletion of used diesel oils. The assumption has been that the difference between the ash of the used oil and that of the new oil is related to the amount of detergent consumed in service. Here again, results may be misleading. Consumption of the additive does not ordinarily mean that it has been disposed of, but that its effectiveness has been exhausted in the performance of its function. The metallic elements may still be present and may appear in the residue in the same concentration as in the new oil.

Sulfated ash of used diesel oils has significance only of a very general nature. If it runs higher than that of a new oil, contamination with dirt or wear metals is suspected, and further analysis is required to identify the foreign material. If sulfated ash runs low, it may be attributed to faulty engine operation or a mechanical defect. With gasoline engines, a high sulfated ash may be caused by the presence of lead derived from the fuel.

AUTO-IGNITION TEMPERATURE

ASTM D 2155

All petroleum products will burn and, under certain conditions, their vapors will ignite with explosive force. For this to happen, however, the ratio of product vapor to air must be within certain limits.

When exposed to air, a certain amount of the liquid product evaporates, establishing a certain vapor-to-air ratio. As the temperature of the liquid increases, so does the evaporation, and thus the vapor/air ratio. Eventually a temperature is reached at which the vapor/air ratio will support combustion if an ignition source, such as a spark or flame, is present. This is the flash point of the product.

If no ignition source is present, as the temperature increases above the product's flash point, a temperature is reached at which the product will ignite spontaneously, without any external source of ignition. This temperature is the auto-ignition temperature of that fluid.

The auto-ignition temperature of a liquid petroleum product at atmospheric pressure is determined by the standard ASTM method D 2155 (which replaces the older ASTM D 286, discontinued in 1966).

Significance of Results

The auto-ignition temperature of a petroleum product is primarily significant as an indication of potential fire and explosion hazards associated with the product's use. The auto-ignition temperature may be used as a measure of the relative desirability of using one product over another in a high-temperature application. It is necessary to use a petroleum product with an auto-ignition temperature sufficiently above the temperature of the intended application to ensure that spontaneous ignition will not occur. Auto-ignition temperature thus places one—but by no means the only—limit on the performance of a product in a given application.

The auto-ignition temperature under a given set of conditions is the lowest tem-perature at which combustion of a petroleum product may occur spontaneously, without

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an external source of ignition. It is not to be confused with the flash point of a product, which is the lowest temperature at which a product will support momentary combustion, in the presence of an external ignition source.

The auto-ignition temperature of a product is a function of both the characteristics of the product and conditions of its environment. For example, the auto-ignition temperature of a substance is a function of such things as the pressure, fuel-to-air ratio, time allowed for the ignition to occur, and movement of the vapor-air mixture relative to the hot surface of the system container. Consequently, the auto-ignition temperature may vary considerably depending on the test conditions.

For a given product at atmospheric pressure, the auto-ignition temperature is always higher than the flash point. In fact, as a general rule for a family of similar compounds, the larger the component molecule, the higher the flash point, and the lower the auto-ignition temperature.

However, as the pressure of the system is increased, the auto-ignition temperature decreases, until a point is reached, usually at a pressure of several atmospheres, at which the auto-ignition temperature of a product under pressure may be less than the flash temperature of the product at atmospheric pressure. Thus, concern for the auto-ignition of a product increases as the pressure on the system increases.

As a general rule, the auto-ignition temperatures of many distillate products with similar boiling ranges can be related to hydrocarbon type. For example, aromatics usually have a higher auto-ignition temperature than do normal paraffins with similar boiling range. The auto-ignition temperatures of isoparaffins and naphthalenes normally fall somewhere in between those of the aromatics and normal paraffins.

However, care should be used in attempting to extend this guideline. For example, increasing the aromatics content of a lube or hydraulic oil tends to reduce the auto-ignition temperature of the oil. Conversely, increasing the aromatic nature of a solvent tends to increase the auto-ignition temperature of the solvent.

BIODEGRADATION AND ECOTOXICITY

Biodegradation is the breakdown of a substance, e.g., hydraulic fluid, by living organisms into simpler substances, such as carbon dioxide (CO_2) and water. Most standard test methods for defining the degree of biodegradation of a substance use bacteria from a wastewater treatment system as the degrading organisms. This provides a relatively consistent source of bacteria, which is important, since the bacteria are the only variable in the test other than the test substance itself. A term that can be roughly defined as the opposite of biodegradability is persistence. A product is persistent if it does not degrade, or if it remains unchanged for long periods of time, i.e., years, decades.)

There are many tests for biodegradation. Depending on the test design, it can measure primary biodegradability or ultimate biodegradability. Primary biodegradability is a measure of the loss of a product, but it does not measure the degree of degradation, i.e., partial or complete (to CO_2 and water), or characterize the by-products of degradation. It merely determines the percentage of the product that disappears over the term of the test

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or, conversely, determines the time required to reach a certain percentage of loss. A popular primary biodegradation test in use today is the CEC-L-33-A-94, which measures disappearance of the test product and relates that to a biodegradation level. The assumption in this test is that all of the product that has disappeared is completely biodegraded. In actuality, this may not be the case, because the test does not measure *complete biodegradation*, but only the *loss* of the original product.

Ultimate biodegradability describes the percentage of the substance that undergoes complete degradation, i.e., degrades to CO_2 and water over the length of the test or, conversely, describes how long it takes to achieve a specified percentage of degradation. Two tests that are designed to measure ultimate biodegradability are the Modified Sturm Test (OECD 301B) and the EPA Shake Flask Test, both of which quantify CO_2 generated over 28 days (a standard test duration).

Thus, the terms primary and ultimate describe the extent of biodegradation. The rate of biodegradation is defined by the term ready biodegradation. A product is considered to be readily biodegradable if shown to degrade 60-70%, depending on the test used. Only a few tests measure ready biodegradability. The more commonly used include: the Modified Sturm Test (OECD 301 B); the Manometric Respirometry Test (OECD 301 F); and the Closed Bottle Test (OECD 301 D).

The final term to discuss here is inherent biodegradability. A product is considered inherently biodegradable if shown to degrade greater than 20%. However, unlike ready biodegradation tests, which run a specified 28 days, tests for inherent biodegradation have no defined test duration and are allowed to proceed as long as needed to achieve 20% degradation, or until it is clear that the product will never biodegrade to that extent. In the latter case, the product is then considered persistent. Evaluating a substance's environmental toxicity (ecotoxicity) can involve examining its effect on growth, reproduction, behavior, or lethality in test organisms. In general, ecotoxicity is measured using aquatic organisms like fish, aquatic insects, and algae. The most common endpoint for expressing aquatic toxicity in the laboratory is the LC_{50}, which is defined as the lethal concentration (LC) of a substance that produces death in 50% of the exposed organisms during a given period of time. Ecotoxicity data, properly developed, understood, and applied, are useful for evaluating the potential hazard of a material in the environment. Some of the most commonly used organisms for aquatic toxicity studies include rainbow trout, mysid shrimp, daphnids (water fleas), and green algae.

Significance of Test Results

Both the test method and the intended use of the data must be considered when evaluating the biodegradability and environmental toxicity of a product. Data from different test methods are generally not comparable, and data developed on different products by different labs should be evaluated with strict caution by experts in the field.

Comparable biodegradation data should be developed using a consistent inoculum (bacteria) source, and in the same time frame, due to the variation of bacterial populations over time. As with most laboratory test procedures, results cannot be directly extrapolated to natural settings.

Similarly, for environmental toxicity tests, comparative data should be developed

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using the same test procedures and the same organisms. Exposures experienced in the laboratory will not be replicated in nature. The natural environment is a large dynamic ecostructure, while the laboratory environment is static and limited in size. Further, if a contaminant enters a natural aquatic system, the event will most likely be random in concentration and frequency, unlike the laboratory environment, which depends on constant, measured contamination.

Biodegradation and environmental toxicity data help us to begin to better understand how to protect the world around us. There are discrete, clearly defined methods for testing products for environmental toxicity, and there are many different test methods for evaluating a product's potential persistence in the environment. However, at this time, in the United States, there is no standard set of universally accepted test procedures defined by government or industry to measure the environmental performance of a product.

Standard biodegradability and environmental toxicity tests are very simplistic in their approach, and the usefulness of the data is generally limited in scope. The test systems typically used will never be able to consider the myriad variables that occur in the environment. In order to truly evaluate the "environmental friendliness" of a product, other investigative approaches, such as Life Cycle Assessment, in which manufacturing, delivery, useful life, and disposal undergo equal scrutiny, should be considered. This is particularly critical when comparing different classes of products, e.g., mineral oil-based versus vegetable oil-based hydraulic fluids.

Too much emphasis should not be placed on the quantitative results from these tests. Environmental studies cannot merely be represented by the simple numerical values that are often used to support claims regarding the "friendliness" of a product. Rather, they need to be understood in the context within which they were developed, i.e., how and why the tests were done. If not considered in that limited context, the information could improperly represent the "friendliness" or "unfriendliness" of a product.

In summary, biodegradation and environmental toxicity test results are not directly comparable in the same way as tests for physical characteristics, such as viscosity. Finally, although they are very important in the overall evaluation of a product, they represent only part of the data important to a product's complete evaluation.

CLOUD POINT

ASTM D 2500

If chilled to sufficiently low temperature, distillate fuels can lose their fluid characteristics. This can result in loss of fuel supply. The time when cold weather causes fuel stoppages is precisely the time when fuel is needed most in residential and commercial heating units. Diesel powered equipment of all kinds is subject to failure due to poor low-temperature operability of the fuel. Consequently, it is generally necessary to know how cold a fuel can become before flow characteristics are adversely affected.

The most important indication of low-temperature flow characteristics of distillate fuels is *cloud point*. This is the temperature at which enough wax crystals are formed to give the fuel a cloudy appearance. (It should not be confused, however, with the turbid

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appearance that is sometimes caused by water dispersed in the fuel.)

Because of the effects of some additives on wax crystal formation, cloud point alone should not be used as an absolute minimum operating temperature. Some of these additives have been shown to lower the minimum operating temperature for specific fuels without affecting the base fuel's cloud point.

Significance

The cloud point of a distillate fuel is related to the fuel's ability to flow properly in cold operations. Some additives may permit successful operation with fuels at temperatures below their cloud points; however, for distillate fuels without additives, clogging of filters and small lines may occur due to wax crystal formation at temperatures near the fuel's cloud point.

COLOR SCALE COMPARISON

Several scales are used to measure color of petroleum products. Approximate conversion and comparison of the more common color scales can be accomplished through use of charts that are available from lubricant suppliers.

Color and Color Tests

Color is a term that is often misunderstood because it is a complex aggregate of human values and physical quantities. No two people have quite the same conception of color when it is allowed to assume its broader meanings. Hue, intensity, tone, purity, wavelength, opacity, and brightness are all directly or indirectly associated with color. It would be extremely difficult to depict mathematically all of these dimensions in a single index. Most attempts to define color do so in terms of only one or two factors, and any meaningful discussion of this index must be strictly confined to the dimensions it is able to represent.

Most of the color tests upon which these scales are based involve the same basic procedure. Light is transmitted simultaneously through standard colored glasses (or other standard reference material) and a given depth or thickness of the sample. The two light fields are compared visually and adjustments are made until a match is obtained. In some test, the volume of the sample is varied until the two fields match (as in the Saybolt test); in others, the light transmitted through a given depth or thickness of the sample is matched by using a series of glasses (as in the ASTM test). When the operator obtains a match, a color value is recorded. This color value corresponds to a point on the color scale associated with the particular color test.

These tests, by definition, involve only two qualities of the transmitted light—appearance, as compared with a standard, and intensity. These two dimensions are not sufficient to describe completely the color of the sample, and should be used only to indicate uniformity and freedom from contamination.

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COMPOSITION ANALYSIS OF PETROLEUM HYDROCARBONS

The analysis of a petroleum hydrocarbon involves the identification or characterization of various components of the substance. This can be accomplished through a variety of techniques. If the amount of information required is great, the analysis can be an extremely complex undertaking. For example, the American Petroleum Institute Project No. 6, an analysis of a single petroleum sample, continued for about 25 years.

The kind of analyses used in quality control and routine laboratory inspections of petroleum products are much faster, of course. These short-cut methods can be carried out in a variety of ways, using different test procedures and different types of instruments. The choice of method depends upon the nature of the substance to be analyzed, and upon the type of information required.

Types of Analysis

The short-cut methods of analysis can generally be classified as either carbon-type or molecular-type. A carbon-type analysis is run when the distribution of the different sizes of molecules—as indicated by the number of carbon atoms in the nucleus—is required. For example, percentages of C_1, C_2, etc. molecules present in the substance can be determined by such an analysis. A molecular-type analysis is run when the object is to characterize the components according to the chemical *arrangement* of their molecules. Since there are several different ways of classifying the chemical structure of hydrocarbons, several different approaches are possible in molecular-type analyses. For example, a molecular-type analysis could be used to determine the relative percentages of the naphthenic, paraffinic, and aromatic components. Another analysis might simply determine the proportions of saturated and unsaturated compounds present.

General Methods and Instrumentation

The analysis of petroleum hydrocarbons is accomplished through use of a variety of instruments and techniques. The most common techniques go by such names as chromatography, mass spectrometry, ultraviolet and infrared absorption analysis, and precipitation analysis, according to the physical principle upon which each is based.

Chromatography is an analytical technique involving the flow of a gas or liquid, together with the material under analysis, over a special porous, insoluble, sorptive medium. As the flowing phase passes over the stationary phase, different hydrocarbon components are adsorbed preferentially by the medium. With some types of chromatography, these components are desorbed through a similar process, and they leave the chromatographic column in distinct individual patterns. These patterns can be detected and recorded, and with proper interpretation can provide an extremely accurate means of determining composition. Chromatography is used in both carbon-type and molecular-type analyses. There are a number of chromatographic methods, each named according to the technique of analysis. *Gas chromatography* refers to the general method that uses a gas as the flowing, or mobile, phase; *gas liquid chromatography*, a more specific term, describes the techniques of using gas as the flowing phase and a liquid as the stationary phase; etc.

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Silica gel analysis is a liquid chromatographic method that also involves physical separation of the components of a substance. The technique is based on the fact that polar compounds are adsorbed more strongly by silica gel than are non-polar saturated compounds. A sample of material under test is passed through a column packed with silica gel. Alcohol, which is more strongly adsorbed than any hydrocarbon, follows the sample through the column, forcing the hydrocarbons out—saturates first, unsaturated compounds next, then aromatic compounds. Small samples of the emerging material are taken periodically, and the refractive index of each sample is measured. From this information, relative percentages can be determined. (Clay/silica gel analysis, a method designed for rubber process oils, uses both activated clay and silica gel to determine the proportion of asphaltene, aromatic, saturated, and polar compounds present.)

Fluorescent indicator analysis (FIA) is a refinement of silica gel analysis in which a mixture of fluorescent dyes is placed in a small layer in the silica gel column. The dyes separate selectively with the aromatics, olefins, and saturates in the sample. Under ultraviolet light, boundaries between these different fractions in the column are visible; the amount of each hydrocarbon-type present can be determined from the length of each dyed fraction.

Mass spectrometry identifies the components of a substance by taking advantage of the difference in behavior exhibited by molecules of different mass when subjected to electrical and magnetic fields. A particle stream of the test material is first ionized, then directed in a curved path by a combination of the electrical and magnetic fields. The heavier ions, having greater inertia, tend toward the outside of the curve The stream of particles is therefore split up into a "mass spectrum"—they are distributed across the path according to their masses. This differentiated stream is played across a detecting slot on the "target," and a record of the analysis is thus made. (When the target is a photographic plate, the instrument is referred to as a Mass "Spectroscope").

As might be expected, the Mass Spectrometer is most useful, at least for hydrocarbon analysis, in the determination of carbon-number distributions. However, because various types of material show distinct spectral patterns, the Mass Spectrometer is also used in molecular-type analysis.

Ultraviolet (UV) Absorption Analysis is a method in which the amount and pattern of ultraviolet light absorbed by the sample is taken as a "fingerprint" of the components. The analysis is carried out through use of a spectrophotometer, which measures the relative intensities of light in different parts of a spectrum. By comparing the UV-absorbance pattern of the test sample with patterns of known material, components of the sample may be characterized. Infrared (IR) Analysis is a similar method but utilizes a different radiation frequency range.

Precipitation Analysis is used primarily in the characterization of rubber process oils. Components are identified on the basis of their reaction with varying concentrations of sulfuric acid. Hydrocarbons are separated into asphaltenes, polar compounds, unsaturated compounds (which are further separated into two groups, First Acidiffins and Second Acidiffins), and saturated compounds.

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CONSISTENCY OF GREASE (PENETRATION)

See "Grease Consistency."

COPPER STRIP CORROSION

ASTM D 130

Many types of industrial equipment have parts of copper or copper alloys. It is essential that any oil in contact with these parts be non-corrosive to them.

Though modern technology has made great progress in eliminating harmful materials from petroleum oils, corrosion is still a possibility to be considered. Certain sulfur derivatives in the oil are a likely source. In the earlier days of the petroleum industry the presence of active sulfur might have been attributable to inadequate refining. Today, however, practical methods have been developed to overcome this problem, and straight mineral oils of high quality are essentially free of corrosive materials.

On the other hand, certain oil additives, such as some of the emulsifying and extreme pressure (EP) agents, contain sulfur compounds. In the higher-quality oils, including those for moderate EP conditions, these compounds are of a type that is harmless to copper. For the more severe EP applications, however, chemically active additives are required for the prevention of scoring and seizure. Though oils containing these additives may not be desirable in the presence of copper or copper alloys, they are indispensable to many applications involving steel parts. Automotive hypoid rear axles are an example of this type of application.

To evaluate the corrosive properties of oils to copper—also to check them for active sulfur-type EP additives—the copper strip corrosion test is a widely accepted procedure. This test—described under the ASTM test method D 130—is applicable to the determination of copper-corrosive properties of certain fuels and solvents as well it is not to be confused, however, with tests for the rust-inhibiting properties of petroleum oils. The copper strip test evaluates the copper-corrosive tendencies of the oil itself—not the ability of the oil to prevent corrosion from some other source.

Significance

In the lubrication of bronze bushings, bearings that contain copper, and bronze wheels for worm-gear reduction units, corrosive oils must be carefully avoided. Because of the use of bronze retainers, manufacturers of anti-friction bearings insist on non-corrosive oils for their products. Hydraulic fluids, insulating oils, and aviation instrument oils must also be non-corrosive. In the machining of non-ferrous metals, moreover, cutting fluids must be of a non-corrosive type. The copper strip corrosion test helps to determine the suitability of these oils for the type of service they may encounter. In addition, it may help to identify oils of the active chemical type formulated for severe EP application.

This test may serve also in the refinery to check finished products for conformity with specifications. It may be applied, too, to solvents or fuels for assurance that these products will not attack cuprous metals with which they come in contact. In addition, there are certain special tests for corrosiveness, including the silver strip corrosion test of

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diesel lubricants. This test is applicable to crankcase oils for engines with silver bearing metals.

In conducting the copper strip corrosion test, there are 3 variables that may affect test results:

1. time of exposure of the copper to the sample
2. temperature of the sample
3. interpretation of the appearance of the exposed sample.

It is reasonable to expect that these variables will be applied in such a way as to reflect the conditions to which the product is to be subjected.

There is nothing to be gained, for example, by testing the oil at 212°F if test results at 122°F give better correlation with actual service conditions. If service conditions are more severe, however, test results at the higher temperature may give a more reliable indication of the oil's performance characteristics. Similarly, selection of the critical ASTM classification must be based on experience gained in the type of service for which the product is formulated. A dark tarnish (Classification 3, Table 3-1) is wholly acceptable, for example, where it has been shown that this degree of copper discoloration is associated with safe performance of the tested product. The flexibility of the copper strip test makes it adaptable to a wide range of products and end uses.

Table 3-1. ASTM copper strip classification.

Classification	Designation		Description
1	Slight Tarnish	1a	Light orange, almost the same as a freshly polished strip
		1b	Dark orange
2	Moderate Tarnish	2a	Claret red
		2b	Lavender
		2c	Multi-colored with lavender blue and/or silver overlaid on claret red
		2d	Silvery
		2e	Brassy or gold
3	Dark Tarnish	3a	Magenta overcast on brassy strip
		3b	Multi-colored with red and green showing (peacock), but no gray
4	Corrosion	4a	Transparent black, dark gray or brown with peacock green barely showing
		4b	Graphite or lusterless black
		4c	Glassy or jet black

For each of the 4 ASTM designations, there are two or more standards considered to cover the same degree of tarnish or corrosion. Differences in the type of chemical reaction produce differences in discoloration. Even a completely non-corrosive oil will alter the appearance of the freshly polished strip.

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DEMULSIBILITY

ASTM D 1401 and ASTM D 2711

In the petroleum industry, the term emulsion usually applies to an emulsion of oil and water. Though mutually soluble only to a slight degree, these substances can, under certain circumstances, be intimately dispersed in one another to form a homogeneous mixture. Such a mixture is an oil/water emulsion, and it is usually milky or cloudy in appearance.

Commercial oils vary in emulsibility. A highly refined straight mineral oil resists emulsification. Even after it has been vigorously agitated with water, an oil of this type tends to separate rapidly from the water when the mixture is at rest. Emulsification can be promoted, however, by agitation and by the presence of certain contaminants or ingredients added to the oil. The more readily the emulsion can be formed and the greater its stability, the greater the emulsibility of the oil. Some products, such as soluble cutting fluids, require good emulsibility and are formulated with special emulsifying agents.

With many other products, however, such as turbine oils and crankcase oils, the opposite characteristic is desired. To facilitate the removal of entrained water, these products must resist emulsification. The more readily they break from an emulsion, the better their demulsibility.

Two tests for measuring demulsibility characteristics have been standardized by the ASTM. The older of the two is ASTM method D 1401, which was developed specifically for steam turbine oils having viscosities of 150-450 Saybolt seconds at 100°F. It can be used for oils of other viscosities if minor changes in the test procedure are made. This method is the one recommended for use with synthetic oils.

The second method, ASTM D 2711 is designed for use with R&O (rust and oxidation inhibited) oils. It can also be used for other types of oils, although minor modifications are required when testing EP (extreme pressure) oils.

Significance of Demulsibility Tests

In many applications, oil is exposed to contamination by water condensed from the atmosphere. With turbine oils, exposure is even more severe, since the oil tends to come in contact with condensed steam.

Water promotes the rusting of ferrous parts and accelerates oxidation of the oil. For effective removal of the water, the oil must have good demulsibility characteristics.

Steam cylinder oils that serve in closed systems require good demulsibility for the opposite reason: to facilitate removal of oil from the condensate, so that oil is kept out of the boiler. Hydraulic fluids, motor oils, gear oils, diesel engine oils, insulating oils, and many similar petroleum products must resist emulsification. Oil and water must separate rapidly and thoroughly.

Either of the ASTM methods is suitable for evaluating the demulsification properties both of inhibited and uninhibited oils. However, correlation with field performance is difficult. There are many cases where the circulating oil is operating satisfactorily in the field, but fails the demulsibility tests in the laboratory. Hence, it must be recognized that these laboratory test results should be used in conjunction with other facts in evaluating an oil's suitability for continued service.

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DENSITY

Density is a numerical expression of the mass-to-volume relationship of a substance.

Density is important in volume-to-mass and mass-to-volume calculations, necessary in figuring freight rates, fuel loads, etc. Although it is not directly a criterion of quality, it is sometimes useful as an indicator of general hydrocarbon type in lubricants and fuels. For a given volatility, for example, aromatic hydrocarbons have a greater density than paraffins, naphthenic hydrocarbons usually being intermediate. Density data may also be used by manufacturers or their customers to monitor successive batches of a product as a check on uniformity of composition.

In the SI system (see the introduction) the official unit for density is kilograms/cubic meter (kg/m^3 at 15.C. Units formerly used were *density* in kilograms/cubic decimeter (kg/dm^3) at 15°C, *specific gravity* 60/60°F (mass per unit volume compared with that of water at the same temperature), and *API gravity* at 60°F (an arbitrary scale calibrated in degrees). An API gravity/specific gravity/density conversion chart appears in the appendix to this book.

Density may be determined by ASTM method D 1298, using a hydrometer graduated in units of density. Tables are available for conversion of observed density to that at 15°C.

See also Gravity.

DIELECTRIC STRENGTH

ASTM D 877 and D 1816

A dielectric is an electric insulating material, one that opposes a flow of current through it. There are two properties that contribute to this characteristic. One is resistivity, the specific resistance that a dielectric offers under moderate conditions of voltage. The other is dielectric strength, the ability to prevent arcing between two electrodes at high electric potentials. Though the two properties are not directly related, it so happens that commercial insulating materials of high dielectric strength also possess adequate resistivity. In the insulation of high-voltage electrical conductors, therefore, it is ordinarily dielectric strength that is of the greater concern.

Petroleum oil is an excellent dielectric and is used extensively in electrical equipment designed to be insulated with a liquid. Among the advantages that oil offers over solid insulation are the abilities to cool by circulation and to prevent corona. Corona is the result of ionization of air in the tiny voids that exist between a conductor and a solid insulating wrapper. Corona is destructive to certain types of solid insulation. By filling all of the space around a conductor, insulating oil eliminates the source of corona. Oil also has the high dielectric strength that good insulation requires.

At normal voltage gradients, conduction of electric current through a dielectric is negligible. The dielectric lacks the free charged particles that a conductor must have. If the voltage impressed on the dielectric is increased, however, the material becomes more

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highly ionized. Ions thus produced are free charged particles.

If a high enough voltage is applied, ions are produced in sufficient concentration to allow a discharge of current through the dielectric, and there is an arc. The minimum voltage required for arcing is the breakdown voltage of the dielectric incurred under the circumstances involved. When the dielectric break down, it undergoes a change in composition that permits it—temporarily, at least—to conduct electricity.

The magnitude of the breakdown voltage depends on many factors, such as the shape of the electrodes and the thickness and dielectric strength of the insulation between them. In accordance with the ASTM method D 877 or D 1816, the dielectric strength of an insulating oil is evaluated in terms of its breakdown voltage under a standard set of conditions. Because of the marked effect of contamination on test results, special care must be exercised in obtaining and handling the sample. The sample container and test cup must be absolutely clean and dry, and no foreign matter must come in contact with the oil.

In either case, the voltage noted at the specified end point is the breakdown voltage of the respective sample.

Significance of Test Results

Insulating oils find wide application in transformers, cables, terminal bushings, circuit breakers, and similar electrical equipment. Depending upon the installation the purpose of these oils may be to prevent electrical leakage and discharge, to cool, to eliminate corona effects, or to provide any combination of these functions. High dielectric strength is obviously an important insulating-oil property.

When new, a carefully refined petroleum oil can be expected to exhibit a high natural dielectric strength suitable for any of the conventional insulating purposes. Other properties of the oil, such as oxidation resistance, are therefore of greater significance.

In service, however, the oil eventually becomes contaminated with oxidation products, carbon particles, dirt, and water condensed from atmospheric moisture. Water is the principal offender. Though small quantities of water dissolved in the oil appear to have little influence on dielectric strength, *free* water has a pronounced effect. The dispersion of free water throughout the oil is promoted, moreover, by the presence of solid particles. These particles act as nuclei about which water droplets form. Dielectric strength is impaired also by dirt and oxidation sludges that may accumulate in the oil.

A relationship exists, therefore, between a drop in dielectric strength and the deterioration of an oil in service. Dielectric strength thus suggests itself as a method of evaluating the condition of a used insulating oil. In this application, a significant drop in dielectric strength may indicate serious water contamination, oxidation, or both.

If water is the only major contaminant, the oil can generally be reclaimed by drying. But, if the drop in dielectric strength is attributable to oxidation, the oil may already have deteriorated beyond a safe limit. By itself, therefore, dielectric strength is not ordinarily considered a sufficiently sensitive criterion of the suitability of a batch of oil for continued service. Power factor, neutralization number, and interfacial tension are test values that have found greater acceptance for this purpose.

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DILUTION OF CRANK CASE OILS

ASTM D 322

Excessive crankcase dilution is associated with faulty operation of an internal combustion engine. It is caused by the seepage of raw and partially burned fuel from the combustion chamber past the piston into the crankcase, where it thins the crankcase oil. It is often desirable to know the extent to which a used oil has been diluted in this way. For motor oils from gasoline engines, dilution may be evaluated by the ASTM method D 322.

The procedure is to measure the percentage of fuel in the sample by removing the fuel from the oil. Since the fuel is considerably more volatile than the oil, the two can be separated by distillation.

To lower the distillation temperature and to make the test easier to run, a relatively large amount of water is added to the sample. Since the water and the sample are immiscible, the boiling point of the mixture is, at any instant, appreciably lower than that of the sample alone.

Because of its substantially higher volatility, the fuel is, to all intents and purposes, evaporated before the oil. A mixture of fuel vapor and water vapor passes into a condenser and is converted back to liquid. The fuel, which is lighter, floats on top of the water in a graduated trap. Here, the volume of condensed fuel can be observed before any significant distillation of the oil begins.

Significance of Results

This test for crankcase dilution is applicable to used motor oils from gasoline engines. Excessive dilution, as determined by test, is harmful in is own right, as well as being indicative of faulty engine performance.

In the first place, dilution is an obvious source of fuel waste. Another effect is to reduce the viscosity of the oil, which may seriously impair its lubricating value. A diluted oil may lack the body required to prevent wear, and it may not make a proper seal at the piston rings. Pistons and cylinders are especially vulnerable, since the oil on their wearing surfaces is subject to the direct washing action of the raw fuel.

Fuel may also reduce the oil's oxidation stability and may raise the oil level in the crankcase. An abnormally high level causes an increase in oil consumption and gives false readings as to the actual amount of lubricant present. Failure of a motor oil to lubricate as it should maybe directly attributable to dilution.

As an indication of faulty performance, excessive crankcase dilution may be the symptom of an unsuitable fuel. If the fuel's volatility characteristics are too low, the fuel does not vaporize properly, and combustion is incomplete. The unburned portion of the fuel finds is way into the crankcase.

A similar effect may be produced, however, by incorrect operation or poor mechanical condition of the engine:

Too rich a fuel mixture—maladjustment of the carburetor or excessive choking may admit more fuel to the combustion chamber than can be burned with the amount of air present.

Too low an engine temperature—defective temperature control or short operating pe-

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riods may keep the engine too cold for proper vaporization.

Inadequate breathing facilities—insufficient venting of the crankcase vapors may interfere with normal evaporation of the fuel from the crankcase. With older cars, the trouble may be caused by stoppage of the crankcase breather. On cars built after 1963, the positive crankcase ventilation system may be at fault.

Worn pistons, rings, or cylinders—excessive clearance between the pistons or rings and the cylinder walls facilitates the seepage of fuel into the crankcase.

Any of the deficiencies indicated by excessive crankcase dilution can be expected to jeopardize satisfactory engine performance.

With diesel engines, there is not the spread in volatility characteristics between fuel and lubricating oil that there is with gasoline engines. For this reason, there is no simple test for the crankcase dilution of diesel engine. The closest approximation is made by noting the reduced viscosity of the used oil as compared with that of the new oil and estimating what percentage of fuel dilution would cause such a viscosity reduction.

DISTILLATION

A chemically pure hydrocarbon, like any other pure liquid compound, boils at a certain temperature when atmospheric pressure is constant. However, almost all commercial fuels and solvents contain many different individual hydrocarbons, each of which boils at a different temperature. If the petroleum product is gradually heated, greater proportions of the lower-boiling constituents are in the first vapor formed, and the successively higher-boiling constituents are vaporized as the temperature is raised.

Thus, for any ordinary petroleum product, boiling takes place over a range of temperature rather than at a single temperature. This range is of great importance in fuel and solvent applications, and is the property measured in distillation tests.

Several ASTM tests are used for measuring the distillation range of petroleum products. These tests are basically similar, but differ in details of procedure. The following tests are widely used

ASTM D 86-67:	Distillation of Petroleum Products
ASTM D 216-54:	Distillation of Natural Gasoline
ASTM D 850-70:	Distillation of Industrial Aromatic Hydrocarbons
ASTM D 1078-70:	Distillation Range of Lacquer Solvents and Dilutants

Significance of Results

For both fuels and solvents, distillation characteristics are important.

Automotive Gasoline: The entire distillation range is important in automotive fuels. The distillation characteristics of the "front end" (the most volatile portion, up to perhaps 30% evaporated), together with the vapor pressure of the gasoline (see discussion on Vapor Pressure), control its ability to give good cold-starting performance. However, these same characteristics also control its vapor-locking tendency. An improvement in cold-starting can entail a decrease in vapor lock protection.

The temperatures at which 50% and 90% of the fuel are evaporated are indications

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of warm-up characteristics. The lower these points, the better the warm-up. A low 50% point is also an indication of good acceleration. A low 90% point is desirable for completeness of combustion, uniformity of fuel distribution to the cylinders, and less formation of combustion chamber deposits.

Usually the volatility of a commercial gasoline is adjusted seasonally, and also in accordance with the climate in the region into which it is being shipped. In cold weather, a more volatile product is desired to provide better starting and warm-up. In warm weather, less volatility provides greater freedom from vapor lock.

Aviation Gasoline: In general, aviation gasolines have lower 90% points and final boiling points than automotive gasolines, but the significance of the various points on the distillation curve remain the same. A minimum limit on the sum of the 10% and 50% points is normally specified to control carburetor icing characteristics.

Diesel Fuel: Although diesel fuels have much lower volatility than gasoline, the effect of the various distillation points are similar. For example, the lower the initial boiling point for a given cetane number, the better its starting ability, but more chance of vapor lock or idling difficulties. Also, the higher the end point or final boiling point, the more chance there is of excessive smoking and deposits. The mid-boiling point (50% point) is related to fuel economy because, other things being equal, the higher the 50% point, the more Btu content and the better cetane number a diesel fuel possesses.

Burner Fuel: For burner fuels, ease of lighting depends on front end volatility. Smoking depends upon the final boiling point, with excessive smoke occurring if the final boiling point is too high.

Solvents: Many performance characteristics of solvents are related to distillation range. The initial boiling point is an indirect measure of flash point and, therefore, of safety and fire hazard. The spread between IBP and the 50% point is an index of "initial set" when used in a rubber or paint solvent.

The 50% point shows a rough correlation with evaporation rate; the lower the 50% point for certain classes of hydrocarbons, the faster the evaporation. If the dry point and the 95% point are close, there is little or no "tail" or slow-drying fractions. Also, useful indications of good fractionization of a solvent are the narrowness of the distillation range and the spread between IBP and 5% point and between 95% point and the dry point. The smaller the spread, the better.

DROPPING POINT OF GREASE

ASTM D 566 and ASTM D 2265

It is often desirable to know the temperature at which a particular lubricating grease becomes so hot as to lose its plastic consistency. Being a mixture of lubricating oil and thickener, grease has no distinct melting point in the way that homogeneous crystalline substances do. At some elevated temperature, however, the ordinary grease becomes sufficiently fluid to drip. This temperature is called the dropping point and can be determined by the ASTM Method D 566—"Dropping Point of Lubricating Grease" and ASTM Method D2265—"Dropping Point of Lubricating Grease of Wide Temperature Range."

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Significance of Results

Since both these test are held under static conditions, the results have only limited significance with respect to service performance. Many other factors such as time exposed to high temperatures, changes from high to low temperatures, evaporation resistance and oxidation stability of the grease, frequency of relubrication, and the design of the lubricated mechanism are all influences that affect the maximum usable temperature for the grease.

Though both dropping point and consistency are related to temperature, the relationships follow no consistent pattern. The fact that a grease does not liquefy at a particular temperature gives no assurance that its consistency will be suitable at that temperature. However, the dropping point is useful in identifying the grease as to type and for establishing and maintaining bench marks for quality control.

One of the weaknesses of either procedure is that a drop of oil may separate and fall from the grease cup at a temperature below that at which the grease fluidizes. This would then give an erroneous indication of the actual temperature at which the grease becomes soft enough to flow from the cup.

ECOTOXICITY

(See "Biodegradation")

FLASH AND FIRE POINTS—OPEN CUP

ASTM D 92

The flash point and the fire point of a petroleum liquid are basically measurements of flammability. The flash point is the minimum temperature at which sufficient liquid is vaporized to create a mixture of fuel and air that will burn if ignited. As the name of the test implies, combustion at this temperature is only of an instant's duration. The fire point, however, runs somewhat higher. It is the minimum temperature at which vapor is generated at a rate sufficient to *sustain* combustion. In either case, combustion is possible only when the ratio of fuel vapor to air lies between certain limits. A mixture that is too lean or too rich will not burn.

The practice of testing for flash and fire points was originally applied to kerosene to indicate its potentiality as a fire hazard. Since then, the scope has been broadened to include lubricating oils and other petroleum products. Though it has become customary to report flash point (and sometimes fire point) in lubricating oil data, these properties are not as pertinent as they might appear. Only in special instances does a lubricating oil present any serious fire hazard. Being closely related to the vaporization characteristics of a petroleum product, however, flash and fire points give a rough indication of volatility and certain other properties.

The fire point of a conventional lubricating oil is so closely associated with its flash point, that it is generally omitted from inspection data. For the ordinary commercial product, the fire point runs about 50°F above the flash point. Fire and flash points are not to be confused, however, with auto-ignition temperature, which is an entirely different matter.

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Auto-ignition deals, not so much with volatility, as with the temperature necessary to precipitate a chemical reaction—combustion—without an external source of ignition. Though a more volatile petroleum product may be expected to have lower flash and fire points than one that is less volatile, its ASTM auto-ignition temperature is generally higher.

Significance of Test Results

To appreciate the significance of flash point and fire point test results, one must realize what the tests measure. It is necessary to understand how a combustible air-fuel mixture is created.

For all practical purposes, a petroleum liquid does not burn as such, but must first be vaporized. The vapor mixes with the oxygen in the air, and, when sufficient concentration of the vapor is reached, the mixture may be ignited, as by a spark or open flame. The mixture can be ignited only if the concentration of fuel vapor in the air is more than about 1% or less than about 6% by volume. A confined mixture containing more than 6% fuel vapor becomes a practical explosion hazard only if it is vented to admit a greater portion of air.

The significance of flash- and fire-point values lies in the dissimilarity that exists in the volatility characteristics of different petroleum liquids. Even among lubricating oils of comparable viscosity, there are appreciable variations in volatility, and hence in flash and fire point. In general, however, the storage and operating temperatures of lubricating oils are low enough to preclude any possibility of fire. Among the exceptions to this situation are such products as quenching and tempering oils, which come in direct contact with high-temperature metals. Heat-transfer oils, used for heating or cooling, may also reach temperatures in the flash- and fire-point ranges. Similarly, in the evaluation of roll oils, which are applied in steel mills to hot metal sheets from the annealing oven, fire hazard may likewise be a consideration. In many of these cases, however, auto-ignition temperature is of greater significance. At the auto-ignition temperature, as determined by test, fire is not mercy a possibility—it actually occurs spontaneously, i.e., without ignition from any outside source.

Since flash and fire point are also related to volatility, however, they offer a rough indication of the tendency of lubricating oils to evaporate in service. It should be apparent that lower flash and fire points imply a greater opportunity for evaporation loss. The relationship between test results and volatility is by no means conclusive, however. The comparison is distorted by several additional factors, the most important of which is probably the manner in which the oil is produced.

The relationship between flash and fire point, on the one hand, and volatility, on the other, is further distorted by differences in oil type. For a given viscosity, a paraffinic oil will exhibit higher flash and fire point than other types and may be recognized by these test results. Paraffinicity may also be indicated by a high viscosity index or by a high pour point.

Fire and flash points are perhaps of greater significance in the evaluation of used oils. If an oil undergoes a rise in flash or fire point in service, loss by evaporation is indicated. The more volatile components have been vaporized, leaving the less volatile ones behind; so an increase in viscosity is apparent. An excessive increase in viscosity may so alter lubricating properties that the oil is no longer suitable for its intended application.

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If, on the other hand, the flash or fire points drop in service, contamination is to be suspected. This may happen to motor oils that become diluted with unburned fuel. Gasoline or heavier fuels in the crankcase reduce the viscosity of the oil, and bearings and other moving parts may be endangered by excessive thinning of the lubricant. These fuels, being more volatile than the oil, lower the flash and fire points of the mixture. So the flash- or fire-point test on used oils constitutes a relatively simple method for indicating the presence of dilution.

FLASH POINT-CLOSED CUP

ASTM D 56 and D93

All petroleum products will burn, and under certain conditions their vapors will ignite with explosive violence. However, in order for this to occur, the ratio of vapor to air must be within definite limits.

When a liquid petroleum product is exposed to air, some of it evaporates, causing a certain vapor/air concentration. As the temperature of the liquid product is raised, more and more evaporates, and the vapor/air ratio increases. Eventually, a temperature is reached at which the vapor/air ratio is high enough to support momentary combustion, if a source of ignition is present. This temperature is the flash point of the product.

For fuels and solvents, the flash point is usually determined by a "closed cup" method, one in which the product is heated in a covered container. This most closely approximates the conditions under which the products are handled in actual service. Products with flash points below room temperature must, of course, be cooled before the test is begun.

Two closed cup methods of determining flash point are widely used. They differ primarily in details of the equipment and in the specific fields of application. However, the tests are basically similar, and may be grouped together for the purpose of description. The two tests are:

ASTM D 56 Flash Point by Means of the Tag Closed Tester
ASTM D 93 Flash Point by Means of the Pensky-Martens Closed Tester

The former test (Tag) is used for most fuels and solvents, including lacquer solvents and dilutants with low flash points. The latter test (Pensky-Martens) is ordinarily used for fuel oils but can also be used for cutback asphalts and other viscous materials and suspensions of solids.

Significance of Results

For a volatile petroleum solvent or fuel, flash point is primarily significant as an indication of the fire and explosion hazards associated with its use. If it is possible for any particular application to select a product whose flash point is above the highest expected ambient temperature, no special safety precautions are necessary. However, gasoline and some light solvents have flash points well below room temperature. When they are used,

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controlled ventilation and other measures are necessary to prevent the possibility of explosion or fire.

It should be remembered that flash point is the lowest temperature at which a product will support momentary combustion, *if a source of ignition is present*. As such, it should not be confused with auto-ignition temperature, which is the temperature at which combustion will take place spontaneously, *with no external source of ignition*. Products with low flash points often have high auto-ignition temperatures, and vice versa.

FOAMING CHARACTERISTICS OF LUBRICATING OILS

ASTM D 892

Foaming in an industrial oil system is a serious service condition that may interfere with satisfactory system performance and even lead to mechanical damage.

While straight mineral oils are not particularly prone to foaming, the presence of additives and the effects of compounding change the surface properties of the oils and increase their susceptibility to foaming when conditions are such as to mix air with the oils. Special additives impart foam resistance to the oils and enhance their ability to release trapped air quickly under conditions that would normally cause foaming.

The foaming characteristics of lubricating oils at specified temperatures are determined by the standard ASTM method D 892.

Significance

Foaming consist of bubbles that rise quickly to the surface of the oil, and is to be distinguished from air entrainment, consisting of slow-rising bubbles dispersed throughout the oil. Both these conditions are undesirable, and are often difficult to distinguish due to high flow rates and turbulence in the system. These two phenomena are affected by different factors and are considered in separate laboratory tests. The primary causes of foaming are mechanical—essentially an operating condition that tends to produce turbulence in the oil in the presence of air. The current trend in hydraulic oil systems, turbine oil systems, and industrial oil systems of every kind is to decrease reservoir sizes and increase flow rates. This trend increases the tendency for foaming in the oils.

Contamination of the oil with surface-active materials, such as rust preventatives, detergents, etc., can also cause foaming.

Foaming in an industrial oil is undesirable because the foam may overflow the reservoir and create a nuisance, and the foam will decrease the lubrication efficiency of the oil, which may lead to mechanical damage.

Antifoaming additives may be used in oils to decrease foaming tendencies of the oil. However, many such additives tend to increase the air entrainment characteristics of an oil, and their use requires striking a balance between these two undesirable phenomena.

FOUR-BALL WEAR TEST—ASTM D 2266
FOUR–BALL EP TEST—ASTM D 2596

(See "Load Carrying Ability")

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GRAVITY

ASTM D 287

Practically all liquid petroleum products are handled and sold on a volume basis—by gallon, barrel, tank car, etc. Yet, in many cases, it is desirable to know the weight of the product. Gravity is an expression of the weight-to-volume relationship of a product.

Any petroleum product expands when it is heated, and its weight per unit volume therefore decreases. Because of this, gravity is usually reported at a standard temperature, although another temperature may actually have been used in the test. Tables are available for converting gravity figures from one temperature basis to another.

Gravity can be expressed on either of two scales. The "specific gravity" is defined as the ratio of the weight of a given volume of the product at 60°F to the weight of an equal volume of water at the same temperature.

In the petroleum industry, however, the API (American Petroleum Institute) gravity scale is more widely used. This is an arbitrary scale, calibrated in degrees, and related to specific gravity by the formula:

$$\text{API Gravity (degrees)} = \frac{141.5}{\text{specific gravity } 60/60°\text{F}} - 131.5$$

As a result of this relationship, the higher the specific gravity of a product, the lower is its API gravity. It is noteworthy that water, with a specific gravity of 1.000, has an API gravity of 10.0°.

Gravity (specific or API) is determined by floating a hydrometer in the liquid, and noting the point at which the liquid level intersects the hydrometer scale. Corrections must then be made in accordance with the temperature of the sample at the time of test.

Significance of Results

Gravity has little significance from a quality standpoint, although it is useful in the control of refinery operations. Its primary importance is in volume-to-weight and weight-to-volume calculations. These are necessary in figuring freight rates, aircraft and ship fuel loads, combustion efficiencies, etc.

To some extent, gravity serves in identifying the type of petroleum product. Paraffinic products have lower specific gravities (higher API gravities) than aromatic or naphthenic products of the same boiling range. Gravity data may also be used by manufacturers or by their customers to monitor successive batches of these products as a check on uniformity of product composition.

Gravity is important in process applications that depend on differences in gravity of the materials used. For example, petroleum products having higher specific gravities than 1.000 (that of water) are necessary in the field of wood preservation in order to permit separation of the materials involved. The specific gravity range of petroleum products is about 0.6 to 1.05.

Gravity is used in empirical estimates of thermal value, often in conjunction with the aniline point. With the exception of the above applications, gravity should not be used as an index of quality.

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Grease Classification and Testing

Grease Classification

1. Group I—General Purpose Greases
   Greases that are expected to give proper lubrication to bearings whose operating temperatures may vary from −40° C to 121° C (−40° F to 250° F).

2. Group II—High Temperature Greases
   Greases that are expected to give proper lubrication to bearings whose operating temperatures may vary from −18° C to 149° C (0° F to 300° F).

3. Group III—Medium Temperature Greases
   Greases that are expected to give proper lubrication to bearings whose operating temperatures may vary from 0° C to 93° C (32° F to 200° F).

4. Group IV—Low Temperature Greases
   Greases that are expected to give proper lubrication to a bearing whose operating temperature may go as low as −55° C (−67° F) or as high as 107° C (225° F).

5. Group V—Extreme High Temperature Greases
   Greases that are expected to give proper lubrication for comparatively short periods of time where bearing operating temperature may be as high as 232° C (450° F).

The requirements of the five groups of greases cover applications where the lubrication is not affected by extremely heavy loads, high speeds or excessive humidity. The tabulation of test requirements which follows is intended to establish for the five groups of greases those characteristics needed to meet the functional requirements of good rolling bearing greases.

Methods of Testing

Test Requirements	Method
ASTM Penetration Normal Worked	Method ASTM D-217
Oxidation	Method ASTM D-942
Low Temperature Torque	Method ASTM D-1478
Water Resistance	Method ASTM D-1264
Dropping Point	Method ASTM D-566
Evaporation	Method ASTM D-972
Dirt	Method Federal Test Method Standard No. 791-B Method 3005.3
Consistency, Stability	Method ASTM D-217
Abrasive Matter	Method ASTM D-1404
Free Acid and Free Alkali	Method ASTM D-128
Fillers	Method ASTM D-128
Moisture	Method ASTM D-128

Test Requirements

Tests	Group I	Group II	Group III	Group IV	Group V
ASTM Penetration Normal Worked[1]	250 to 350	200 to 300	220 to 300	260 to 320	250 to 310
Oxidation	10* max. drop in 500 hours	10* max. drop in 500 hours	10* max. drop in 500 hours	5* max. drop in 500 hours	5* max. drop in 100 hours at 121° C (250° F)
Low Temperature Torque	1 rev. in max. of 10 sec. at −40° C (−40° F)	—	—	1 rev. in max. of 5 sec. at −55° C (−67° F)	1 rev. in max. of 10 sec. at −40° C (−40° F)
Water Resistance	50% max. loss	—	50% max. loss	20% max. loss	50% max. loss
Dropping Point	149° C min. (300° F)	177° C min. (350° F)	149° C min. (300° F)	149° C min. (300° F)	232° C min. (450° F)
Evaporation	—	—	—	1.5% max. in 22 hours at 121° C (250° F)	4.0% max. in 22 hours at 204° C (400° F)
Dirt Max. Particles per cubic cm	5000/5-20 microns 2000/20-50 microns 50/50-75 microns None over 75 microns	Same as Group I	Same as Group I	Same as Group I	Same as Group I
Consistency Stability: max. ASTM Penetration after 100,000 strokes	Penetration shall not increase more than 100 points and in no case be more than 375	Penetration shall not increase more than 100 points and in no case be more than 350	Penetration shall not increase more than 100 points and in no case be more than 350	Penetration shall not increase more than 100 points and in no case be more than 375	Penetration shall not increase more than 100 points and in no case be more than 375

[1]National Lubricating Grease Institute Code — No. 0: 355-385; No. 1: 310-340; No. 2: 265-295; No. 3: 220-250; No. 4: 175-205

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GREASE CONSISTENCY

ASTM D 217 and D 1403

The consistency of a lubricating grease is defined as its resistance to deformation under an applied force—in other words, is relative stiffness or hardness. The consistency of a grease is often important in determining its suitability for a given application.

Grease consistency is given a quantitative basis through measurement with the ASTM Cone Penetrometer. The method consists of allowing a weighted metal cone to sink into the surface of the grease, and measuring the depth to which the point falls below the surface. This depth, in tenths of millimeters, is recorded as the penetration, or penetration number, of the grease. The softer the grease, the higher its penetration.

The ASTM D 217 method recognizes five different categories of penetration, depending on the condition of the grease when the measurement is made. *Undisturbed* penetration is determined with the grease in its original container. *Unworked* penetration is the penetration of a sample which has received only minimum disturbance in being transferred from the sample can to the test cup. *Worked* penetration is the penetration of a grease sample that has been subjected to 60 double strokes in a standard grease worker (to be described). *Prolonged worked* penetration is measured on a sample that has been worked the specified number of strokes (more than 60), brought back to 77°F, then worked an additional 60 double strokes in the grease worker. *Block* penetration is the penetration of a block grease—a grease hard enough to hold its shape without a container.

All the above penetrations are determined on samples that have been brought to 77°F.

Significance

If a grease is too soft, it may not stay in place, resulting in poor lubrication. If a grease is too hard, it will not flow properly, and either fail to provide proper lubrication or cause difficulties in dispensing equipment. These statements sum up the reasons for classifying greases by consistency. Penetration numbers are useful for classifying greases according to the consistencies required for various types of service, and in controlling the consistency of a given grade of grease from batch to batch.

The National Lubricating Grease Institute has classified greases according to their worked penetrations. These NLGI grades, shown in Table 3-2, are used for selection of greases in various applications.

In comparing greases, worked and prolonged worked penetrations are generally the most useful values. The change in penetration between the 60-stroke value and prolonged worked value is a measure of grease stability. Prolonged worked penetrations should report the amount of working (10,000 and 100,000 strokes are most common) in order to be useful. Unworked penetrations often appear in specifications and in grease product data, but are of limited practical value. No significance can be attached to the difference between unworked and worked penetration. Undisturbed penetration is useful mainly in quality control.

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Table 3-2. NLGI grease grading system.

NLGI Grade	Worked Penetration Range mm/10
000	445-475
00	400-430
0	355-385
1	310-340
2	265-295
3	220-250
4	175-205
5	130-160
6	85-115

INTERFACIAL TENSION

ASTM D 971

Molecules of a liquid have a certain attraction for one another. For some liquids, like mercury, this attraction is very great; for others, like alcohol, it is considerably less. Beneath the surface, the attractive forces are evenly distributed, since a molecule is drawn to the one above or below it as strongly as to the one at its side. But, at the surface, there are no similar molecules over it to attract the liquid upward; so the bonds between the molecules are concentrated in a lateral direction.

The strong mutual attraction between the surface molecules results in a phenomenon known as surface tension, and its effect is like that of a membrane stretched over the liquid face. Surface tension is an appreciable force, as anyone knows who has made the simple experiment of "floating" a needle on the top layer of still water.

Surface tension can be reduced, however, by the introduction of materials that weaken the links between the original molecules. Differences in surface tension can be measured, and these measurements sometimes serve as a guide to the condition of a used oil. Standard procedure for making these measurements is covered by the ASTM method D 971 for conducting the interfacial tension—IFT—test.

The IFT test is one for measuring the tension at the interface between two immiscible liquids: oil and distilled water. Ordinarily, oil and water do not mix, the oil floating on top of the water because it is less dense. At the interface, each liquid exhibits its own surface tension, the molecules of one having no great attraction for those of the other. To break through the interface, it is necessary to rupture the surface tensions both of the water and of the oil. However, if certain contaminants are added to the oil—such as soaps, dust particles, or the products of oil oxidation—the situation is altered. These contaminants are said to be hydrophilic, i.e., they have an affinity for the water molecules—as well as for the oil molecules. At the interface, the hydrophilic materials extend bonds across to the water, so that any vertical linkage between the liquids is strengthened, and

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the lateral linkage is weakened. The interface is less distinct, and the tension at the interface is reduced. The greater the concentration of hydrophilic materials, the less the tension. Since oxidation products tend to be hydrophilic, IFT test results are related to the degree of the oil's oxidation.

Significance of Results

For many purposes, large quantities of petroleum oil remain in service for very long periods. There is good reason, therefore, for checking the extent to which these oils have oxidized to determine their fitness for continued service. In some cases, neutralization number may provide a criterion by which used oils are evaluated. But, by the time the acid neutralization number has undergone a significant rise, oxidation has set in, and acids and sludges may be already formed.

It is felt by many people that the IFT test is more sensitive to incipient oxidation, that it anticipates oxidation before deterioration has reached serious proportions. Since the oxidation of an oil increases at an accelerated rate; an early warning of impending deterioration is advantageous.

The IFT test is frequently applied, therefore, to electric transformer oils, where oxidation is especially harmful. Acids formed by oxidation may attack the insulation, and oxidation sludges interfere with circulation and the cooling of the windings. Because of the importance of good quality, transformer oils may be checked periodically for IFT to determine the advisability of replacement. The critical IFT value is based on experience with the particular oil in service, and testing conditions must be uniform in all respects. Tests for power factor and dielectric strength are also related to the condition of the oil.

For new oils, IFT values have little significance, though they may be used for control purposes in oil manufacture. Additives added to the oil to improve its performance may grossly distort IFT test results, so that they bear no apparent relation to the oil's quality. For this reason, special consideration must be given if attempts are made to evaluate the condition of an inhibited steam turbine oil by the IFT method.

The IFT test itself is an extremely delicate one, and consistent results are not easily obtained. The test should be conducted only by an experienced person, and the apparatus must be scrupulously clean. A minute quantity of foreign matter can cause a tremendous increase in the oil's hydrophilic properties. The sample must be carefully filtered to remove all solid materials, which reduce the IFT value appreciably. Even under the most meticulous conditions, however, good reproducibility is difficult to achieve.

It has been found, moreover, that test results are affected by conditions outside of the laboratory. Prolonged storage of the oil sample may cause a drop in its IFT value; so may exposure to sunlight. Similarly, agitation of the sample may increase its hydrophilic properties, and, if the sample is not tightly sealed against air, there may be an increase in the oxidation products. While indifferent handling practices would not be expected to affect a new oil, they may cause the deterioration of a used oil to appear greater than it actually is.

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LOAD-CARRYING ABILITY

For machine parts that encounter high unit loadings, the lubricant must be capable of maintaining a film that prevents metal-to-metal contact under the extreme pressures involved. Otherwise, scoring of the surfaces and possible failure of the parts will result. Special extreme pressure (EP) lubricants are required for such applications.

Several test machines have been constructed and test procedures established in attempts to approximate closely the conditions a lubricant will meet in field applications. Four widely used tests are the Timken machine, the FZG test, the 4-Ball EP test, and the 4-Ball friction and wear test.

Timken Machine—In the Timken test (ASTM D 2509), a rotating member is brought to bear against a stationary member with lubrication provided by the lubricant under test. The lubricant is evaluated on the basis of its ability to prevent scoring of the metal surfaces. The maximum load that can be applied without scoring is reported as the Timken OK load. The minimum load required to cause scoring is reported as the score load.

In addition to the OK and score loads, the actual pressure at the point of contact is sometimes reported. The area of the wear scar is determined using a Brinnell microscope; the unit loading in megapascals (or in psi) on the area of contact can then be calculated using the OK load.

Only very general conclusions can be drawn from the Timken EP test. Results should be related to additional information about the lubricant, such as antiwear properties, type of additive, and corrosion characteristics. Used in this way, Timken EP results can provide an experienced engineer with valuable information about the performance of one lubricant relative to another. In addition, the Timken EP test is often used in quality control of lubricants whose performance characteristics have already been established.

FZG Test—The FZG test is used in Europe to evaluate EP properties. Two sets of opposing gears are loaded in stages until failure of geartooth surfaces occurs. Results are ported in terms of the number of stages passed. Two standard sets of temperature and gear speed are used and should be stated along with the number of stages passed.

Four-Ball Wear Test and Four-Ball EP Test—Each of the four-ball tests is designed to evaluate a different load-carrying characteristic of lubricating oil or grease. Both use similar equipment and mechanical principles. Four 1/2-inch steel balls are arranged with one ball atop the three others. The three lower balls are clamped together to form a cradle, upon which the fourth ball rotates on a vertical axis.

The four-ball wear test (ASTM D 2266) is used to determine the relative wear-preventing properties of lubricants on sliding metal surfaces operating under boundary lubrication conditions. The test is carried out at specified speed, temperature, and load. At end of a specified period the average diameter of the wear scars on the three lower balls is measured and reported.

Under standardized conditions, the four-ball wear test provides a means for comparing the relative antiwear properties of lubricants. Results of two tests run under differ-onditions cannot be compared, so operating conditions should always be reported. correlation has yet been established with field service, so individual results should

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not be used to predict field performance.

The *four-ball EP test* (ASTM D 2596) is designed to evaluate performance under much higher unit loads than applied in the wear test, hence the designation EP (extreme pressure). The EP Tester is of slightly different design and construction than the Wear Tester. One steel ball is rotated against the other three at constant speed, but temperature is not controlled. The loading is increased at specified intervals until the rotating ball seizes and welds to the other balls. At the end of each interval, the scar diameters are measured and recorded.

Two values from the EP test are generally reported: load wear index (formerly called Mean Hertz Load) and weld point. *Load Wear Index (LWI) is* a measure of the ability of a lubricant to prevent wear at applied loads. *Weld point is* the lowest applied load at which either the rotating ball seizes and then welds to the three stationary balls, or at which extreme scoring of the three stationary balls occurs. It indicates the point at which the extreme pressure limit of the lubricant is exceeded.

The four-ball EP test is used in lubricant quality control, and to differentiate between lubricants having low, medium, or high extreme pressure qualities. Results do not necessarily correlate with actual service and should not be used to predict field performance unless other lubricant properties are also taken into consideration.

For comparison of the capabilities of various lubricants, the results of both four-ball tests should be considered, particularly if additives or grease thickeners are unknown or widely dissimilar. Lubricants with good extreme pressure properties may not be equally effective in reducing wear rates at less severe loads, and conversely.

NEUTRALIZATION NUMBER

ASTM D 664 and D 974

Depending on its source, additive content, refining procedure, or deterioration in service, a petroleum oil may exhibit certain acid or alkaline (base) characteristics. Data on the nature and extent of these characteristics may be derived from the product's neutralization number—or "neut number," as it is commonly known. The two principal methods for evaluating neut number are ASTM D 664 and ASTM D 974. Although respective test results are similar, they are not identical, and any reporting of results should include the method by which they are obtained.

Acidity and Alkalinity

Acidity and alkalinity are terms related to dissociation, a phenomenon of aqueous solutions. Dissociation is a form of ionization, the natural breaking up of some of the molecules into positive and negative ions. If the chemical composition of the aqueous solution is such that it yields more hydrogen ions (positive) than hydroxyl ions (negative), the solution is considered acid; an excess of hydroxyl ions on the other hand results in a solution that is considered to be basic or alkaline. The greater the excess, the more acid or alkaline the solution, as the case may be. If the hydrogen and hydroxyl ions are in equal concentration, the solution is—by definition—neutral.

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Titration

Since acidity and alkalinity are opposing characteristics, an acid solution can be neutralized (or even made alkaline) by the addition of a base. The converse is also true. In either case, neutralization can be accomplished by titration, the gradual addition of a reagent until a specified end point is reached. The amount of acid or base materials in a solution can thus be measured in terms of the quantity of added reagent. Being nonaqueous, however, petroleum oils cannot truly be said to be acid or alkaline. Nevertheless, they can be modified to exhibit these properties by addition of water—plus alcohol to extract oil-soluble acid or alkaline compounds from the sample, and to dissolve them in the water. This principle is utilized in the determination of neutralization number.

pH

Actual acidity or alkalinity, on the other hand, can be expressed in accordance with the pH scale, where zero represents maximum acidity, 14 maximum alkalinity, and 7 neutrality. The pH value of a solution can be determined electrolytically. When two electrodes of different materials are immersed in the solution, a small electric potential (voltage) is generated between them, and the magnitude and polarity of this potential can be related directly to pH value.

Potentiometric Method

The potentiometric method for determining neut number (ASTM D 664) is based on the electrolytic principle, pH, as indicated potentiometrically, is recorded against added reagent. If the initial pH reading of the specially prepared sample lies between 4 and 11 (approximately), the sample may contain weak acids, weak bases, or an equilibrium combination of the two. It may be titrated to one end point with base to yield a total acid number, and then may be titrated to another end point with acid to yield a total base number.

If, on the other hand, the initial pH reading lies below 4 (approximately), the sample may be titrated with base up to this point to yield a strong acid number. It may also be titrated up to 11 (approximately) to yield a total acid number. Similarly, a sample whose initial pH reading lies above 11 (approximately) can be titrated with acid down to is value to yield a strong base number, and it can be titrated down to 4 (approximately) to yield a total base number.

End Points

Titration end points are not at fixed pH readings but at inflections that occur in the curve: reagent versus pH. Whether or not an end point represents a strictly neutral condition is of little significance. With test procedure carefully standardized, the results obtained in reaching an end point can be compared on an equal basis with other results obtained in the same way. A result reported simply as "neut number," moreover, may be assumed to be a total acid number. Although it is not provided for by ASTM procedure, the initial pH reading may also be reported.

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Colorimetric Method

Under the colorimetric method for determining neut number (ASTM D 974), end point is identified by the change of a color indicator. This indicator exhibits one color above a specified pH value, another below. By this means, a total acid or strong base number can be determined with a *p*-naphtholbenzene indicator, while a strong acid number can be determined with a methyl orange indicator. Obviously, however, this method is not suitable for the investigation of dark-colored liquids.

Reporting the Results

Whatever the method, all acid numbers are expressed in milligrams of potassium hydroxide (KOH)—a base—required to "neutralize" a gram of sample. For reasons of uniformity, base numbers, which are obtained by titrating with hydrochloric acid (HC1), are expressed in the same units, the HC1 being converted to the number of KOH units that it would neutralize.

Significance of Results

Because acidity is associated with corrosiveness, there has been a tendency to attribute undesirable properties to an oil that exhibits a high acid number or a low pH reading. This attitude is fostered by the fact that deterioration of an oil in service—oxidation—is ordinarily accompanied by an increase in acid test results. While this attitude is not in actual disagreement with fact, its oversimplification may be conducive to harmful misconceptions.

In the first place, petroleum oil is not an aqueous solution, and conventional interpretations of acidity and alkalinity do not apply. In the second place, the test results, while involving certain acid or alkaline implications, do not distinguish between those that are undesirable and those that are not. The ASTM Standards themselves include the statement that the test "method is not intended to measure an absolute acidic or basic property that can be used to predict performance of an oil under service conditions. No general relationship between bearing corrosion and acid or base number is known."

This is not to say, however, that neut number or pH reading are without significance. They are applied widely and effectively to turbine oils, insulating oils, and many other oils in critical service. With new oils, neutralization test results provide a useful check on consistency of product quality. With used oils, they may serve as a guide to mechanical condition, change in operating conditions, or product deterioration. A rise in acid number and/or a drop in base number or pH reading are generally indicative of increasing oxidation. They may also be related to depletion of an additive, many of which are alkaline.

It is impossible, however, to generalize about the limits to which the neutralization values of an oil in service may safely be allowed to go. Each combination of oil, machine, and type of service follows a pattern of its own. Only through experience with a particular set of conditions can it be determined at what neutralization value an oil is no longer suitable for service.

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OCTANE NUMBER

ASTM D 2699 and D 2700

The octane number of a gasoline is a measure of its antiknock quality; that is, its ability to burn without causing the audible "knock" or "ping" in spark-ignition engines. While octane number is a common term, it is also widely misunderstood, primarily because there are several different methods of measuring this property. Motor Octane Number, Research Octane Number, and Road Octane Number are the three basic procedures. Each assesses antiknock quality of a given fuel under a particular set of conditions.

Octane Number in the Laboratory

In principle, the octane number of a fuel is a numerical expression of its tendency to prevent engine knock relative to a standard fuel. In the laboratory, this quantity is determined through use of the ASTM engine, a special, single-cylinder engine whose operating characteristics can be varied.

The fuel to be rated is first burned in the engine, and the air-fuel mixture is adjusted to produce *maximum knock*, which is measured by a sensing device known as a *knockmeter*. Next, the compression ratio of the engine is varied until a knock intensity of 55 is recorded on the knockmeter. The knock intensity of the test fuel under these conditions is then compared (by referring to charts) to the knock intensities of various reference fuels. The reference fuels are normally blends of two hydrocarbons—iso-octane, which resists knocking, and normal heptane, which knocks severely. Iso-octane is arbitrarily assigned an octane number of 100, while heptane is rated as zero.

The percentage, by volume, of iso-octane in the blend that matches the characteristics of the it fuel is designated as the Octane Number of the fuel. For example, if a blend of 90% isooctane and 10% heptane will match the knock intensities of the "unknown" fuel, under the same conditions, the fuel would be assigned an octane number of 90. (For fuels having octane numbers above 100, the gasoline under test is compared with blends of isooctane and tetraethyl lead, an effective antiknock agent. Such blends can have octane numbers considerably above 100.)

The general test procedure outlined above is the basis for two distinct laboratory methods of determining octane number, *Motor Octane Number* and *Research Octane Number*. Motor Octane Number, ASTM D 2700, is the name given to the octane rating as determined with the ASTM engine and a standard set of operating conditions that became widely known during the 1930's. Research Octane Number, ASTM D 2699, is a more recent method, and is determined under another set of conditions, the chief difference being the slower engine speed. The research method is therefore less severe than the motor method, and most gasolines have a higher octane number by the research method.

Road Octane Number

Laboratory octane ratings do not always provide an accurate prediction of how a fuel will behave in an automobile engine. A more reliable means of predicting antiknock quality is to test the gasolines in automobiles under varying condition of speed and load. There are several methods of determining this rating, which is known as *Road Octane*

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Number; each method compares the test fuel with various blends of iso-octane and heptane.

The Uniontown Procedure, one of the most common Road Octane methods, records the knock intensity at various speeds during acceleration. Knock ratings are recorded, either with instruments or by the human ear. The procedure is repeated, using various blends of iso-octane and heptane, until a reference fuel that produces the same knock characteristics is found. The test fuel is then assigned the same octane number as the reference blend.

The Modified Uniontown Procedure, another common method, depends on the human ear to establish where "trace knock" first occurs. A series of test runs, using various reference fuels of known octane numbers, is first made. For each blend, the spark advance setting that produces trace knock is determined, and the various settings are plotted into a curve. The Road Octane Number of the test fuel can then be assessed by referring to the curve to determine the octane number associated with the spark advance setting that produced trace knock with the test fuel.

Aviation Gasoline Knock Rating

The antiknock level of aviation gasoline is indicated by composite grade numbers, i.e., 80/87, 100/130, 115/145. In each case, the first number is the knock rating determined under conditions of lean air-fuel ratio by ASTM method D 614, while the second number is the rating under the supercharged-rich method, ASTM D 909. Values above 100 are expressed as Performance Numbers, which are related to the number of milliliters of tetraethyl lead in iso-octane.

Significance

Motor Octane Number is normally taken as an indication of a fuel's ability to prevent knocking at high engine speeds, while Research Octane Number measures low-speed knocking tendencies. It is the Road Octane Number, however, that an automobile engine will actually "see" in a given fuel with regard to knock characteristics.

The amount of technical literature devoted to octane numbers is immense, and many correlations exist among the three methods of determining octane numbers which, in the hands of experts, can be meaningful. For the motorist, however, the Road Octane Number of a gasoline offers the most practical prediction of whether the fuel is going to knock in his engine under the conditions to which the car is subjected.

OIL CONTENT OF PETROLEUM WAX

ASTM D 721

A major step in wax refining is the removal of oil; fully refined paraffin waxes usually contain less than 0.5% oil. Therefore, a measure of the oil content of a wax is also an indirect measure of the degree of refinement, and is a useful indicator of wax quality.

The ASTM D 721 test method is based on the low-temperature insolubility of wax in methyl ethyl ketone. A sample of the wax is dissolved in the solvent under heat, the

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solution is cooled to precipitate the wax and then filtered. The oil content of the filtrate is determined by evaporating the methyl ethyl ketone and weighing the residue. By definition, the oil content of a wax is that portion which is soluble in methyl ethyl ketone at −25°F.

Significance of Oil Content

The oil content of a petroleum wax is a criterion of purity and degree of refinement. Highly refined petroleum waxes have high purity and low oil content. This renders them suitable for many applications in the manufacture of drugs, other pharmaceuticals, and food packages. Crude scale waxes are not so highly refined and consequently contain more oil. Such waxes are suitable for applications where some odor or taste can be tolerated, and where higher oil content is permitted.

Oil content of microcrystalline wax can also be determined by ASTM D 721.

OIL SEPARATION IN GREASE STORAGE

ASTM D 1742

ASTM Method D 1742, "Oil Separation From Lubricating Grease During Storage," provides an indicator of the tendency of greases to separate oil while in containers in wage. The separation of a few ounces of oil at the top of a 35-lb container of grease may present less than 1% of the total oil in the grease and is not detrimental to the performance of the product. However, this may produce housekeeping problems as well as cause loss of the user's confidence.

Significance

ASTM states that the test correlates directly with the oil separation that occurs in 35-pound grease pails in storage. The test is also indicative of the separation that may occur in other sizes of containers. This method is not suitable for greases softer than NLGI No. 1 consistency and is not intended to predict the bleeding tendencies of grease under dynamic service conditions.

Due to improved grease technology the problem of grease separation in containers rarely occurs today. Therefore, the relevance of this test to the service performance of modern greases is questionable. The test is primarily of value as a means of assuring batch-to-batch uniformity.

OXIDATION STABILITY—OILS

ASTM D 943

Oxidation is a form of deterioration to which all oils in service are exposed. It is a chemical reaction that occurs between portions of the oil and whatever oxygen may be present—usually the oxygen in the atmosphere. The oxidation of lubricating oils is accelerated by high temperatures, catalysts (such as copper), and the presence of water, acids,

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or solid contaminants. The rate of oxidation increases with time.

Oxidation tends to raise the viscosity of an oil. The products of oxidation are acid materials that lead to depositing of soft sludges or hard, varnish-like coatings. Paraffinic oils characteristically have greater oxidation resistance than naphthenic oils, although naphthenic oils are less likely to leave hard deposits. Whatever the net effect of oxidation, it is undesirable in any oil that lubricates on a long-term basis. Much has been done to improve oxidation resistance by the use of selected base stocks, special refining methods, and oxidation inhibitors. As might be expected, moreover, a great deal of study has been devoted to the means by which oxidation resistance of an oil may be evaluated.

A number of oxidation tests are in use. Some may be better related to a particular type of lubrication service than others. All are intended to simulate service conditions on an accelerated basis. At an elevated temperature, an oil sample is exposed to oxygen or air—and sometimes to water or catalysts—usually iron and/or copper. All of these factors make oxidation more rapid. Results are expressed in terms of the time required to produce a specified effect, the amount of sludge produced or oxygen consumed during a specified period.

One of the more common methods of examining steam turbine oils is the ASTM method D 943. This test is based on the time required for the development of a certain degree of oxidation under accelerated conditions; the greater the time, the higher the oil's rating. Here, oxidation is determined by an increase in the oil's acidity, a property measured by its acid neutralization number. (See discussion on *"Neutralization Number."*)

Significance of Results

Oxidation stability is an important factor in the prediction of an oil's performance. Without adequate oxidation stability, the service life of an oil may be extremely limited. Unless the oil is constantly replaced, there is a serious possibility of damage to lubricated parts. Acids formed by oxidation may be corrosive to metals with which the oil comes in contact. Sludges may become deposited on sliding surfaces, causing them to stick or wear; or they may plug oil screens or oil passages.

Oxidation stability is a prime requisite of oils serving in closed lubrication systems, where the oil is recirculated for extended periods. The higher the operating temperature, the greater the need for oxidation stability, especially if water, catalytic metals, or dirt are present. Resistance to oxidation is of special importance in a steam-turbine oil because of the serious consequences of turbine bearing failure. Gear oils, electric transformer oils, hydraulic fluids, heat-transfer oils, and many crankcase oils also require a high degree of oxidation stability.

Obviously, the ability to predict oxidation life by a test, and to do it with reasonable accuracy is highly desirable. There are certain factors, however, that make reliable test results difficult to obtain. In the first place, the tests themselves are very time-consuming; a method such as ASTM D 913 may require the better part of a year to complete. Prolonged though the test may be, moreover, its duration usually represents but a small fraction of the service life of the oil under investigation. A steam turbine oil, for example, may well last for a decade or more without serious deterioration. It is impossible to reproduce service conditions of this sort in the laboratory with a test even of several hun-

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dred hours' duration. And, in addition to the time factor, there are many other operational variables that cannot be duplicated under test conditions. Results can be distorted also by the presence of certain additives in the oil.

For these reasons, the correlation between oxidation test results and field experience leaves much to be desired. Test results are subject only to broad interpretations. It would be difficult to show, for example, that an oil with a 3000-hour ASTM test life gives better service than an oil with a 2500-hour test life. In evaluating the oxidation stability of an oil, primary consideration should be given to the record that it has established over the years in the type of service for which it is to be used.

OXIDATION STABILITY—GREASES

ASTM D 942 • 1P142, D 1402, and D 1261

The bomb oxidation test was developed in 1938 by the Norma-Hoffman Bearing Corporation. Its purpose was to evaluate the oxidation stability of a grease during the storage of machine parts to which it had been applied. It was not intended to predict her the stability of greases in service or their shelf life in commercial containers.

Method of Evaluation

Oxidation is a form of chemical deterioration to which no petroleum product is immune. Petroleum products vary appreciably in their resistance to oxidation, a property that can be evaluated in many ways for many purposes. In the case at hand, evaluation is related to the quantity of oxygen that reacts with a grease sample during a specified period under standard conditions. The oxidation rate is plotted as pressure drop vs. time, Figure 3-1.

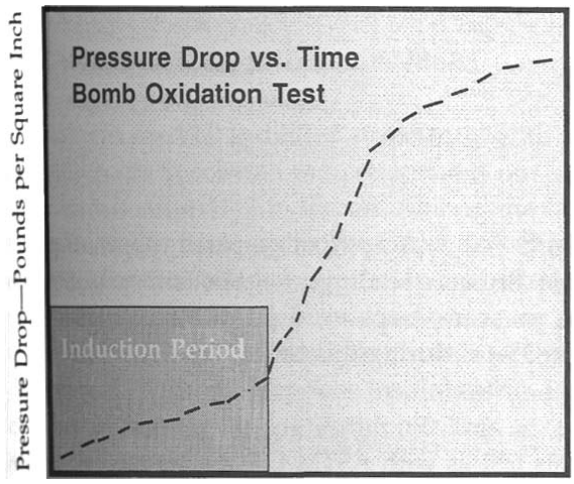

Figure 3-1. When pressure drop is plotted against time, the resulting curve will indicate a period of comparatively slow oxidation followed by a pronounced rise. The relatively flat portion at the beginning represents what is known as the "induction period," a phase during which oxidation is not ordinarily of serious magnitude. For practical reasons, it is not customary to continue the test beyond the induction period, its end being indicated by a sudden rise. Should the test be carried further, however, this rise would eventually taper off again as oxidation becomes complete. In some cases, test results have been expressed in terms of the duration of the induction period.

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ASTM D 942 • 1P142

Procedure

Four grams of the grease to be tested are placed in each of five Pyrex dishes. These samples are then sealed and pressurized with oxygen (at 110 psi) in a heated bomb (210°F). The pressure is observed and recorded at stated intervals. The decrease in oxygen pressure determines the degree of oxidation after a given time period.

Significance of Results

A relationship exists between the pressure lost during this time and the amount of oxygen that has entered into chemical reaction with the grease. However, the drop in pressure is the *net* change resulting from absorption of oxygen and the release of gaseous products by the grease. Thus, this is a basic weakness of the test, since a grease that is being oxidized and at the same time is releasing gaseous products would appear to have greater oxidation resistance than is actually the case. This is a static test and is not intended to predict the stability of grease under dynamic conditions. Nor does it reflect oxidizing influence on bulk quantities in the original container. It more closely represents the conditions in a thin film of grease, as on pre-lubricated bearings or machine parts subjected to extended storage.

Certain machine parts are stored after an application of lubricating grease has been applied. This is particularly true of lubricated-for-life anti-friction bearings, which are greased by the manufacturer and then sealed. It is a common practice to make up these parts in advance and then stockpile them against future requirements.

There is hardly need to point out the damage that can be inflicted by a grease that deteriorates rapidly during this type of storage. The acidity associated with grease oxidation is corrosive to the highly sensitive bearing surfaces, and oxidation deposits may bind the bearing's action even before it has been put in operation. At best, a grease that has undergone significant deterioration in storage can hardly be in a condition to yield the long service life expected of it.

ASTM D 1402

Procedure

This test is run the same as ASTM D 942 is except that prepared copper strips are immersed on edge in each grease sample. Pressure readings are taken at 2-hour intervals over the duration of the test—until the pressure drops to 55 psi or for a specified time period if the pressure hasn't dropped to 55 psi during this time.

Significance of Results

The same limitations exist with this test as with ASTM D 942 since the determination of oxygen absorption rate as an indication of oxidation reaction is affected by the release of gaseous products from the grease.

Results from this test indicate the catalytic effect of copper and its alloys) in accel-

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erating the oxidation of greases under static conditions. The results are not applicable to greases under dynamic conditions or when stored in commercial containers.

ASTM D 1261

Procedure

Each of two Pyrex dishes is filled with 4 grams of grease and a prepared copper strip is partially immersed in each grease sample. The procedure from this point on is the same as in ASTM D 942. At the end of the test time—24 hours—the copper strips are removed, washed, and examined for evidence of discoloration, etching, or corrosion.

Significance of Results

The effect of grease on copper parts of bearing assemblies with which the grease comes in contact is determined from the results of this test. In addition, some indication of the storage stability of greases which are in contact with copper may be found by visual inspection of the grease at the end of the test. The results do not apply to greases in contact with copper under dynamic service conditions.

In spite of the aforementioned limitations, these tests do have significant value. Many concerns find that the bomb test serves as an accurate check on uniformity of grease composition. Though test results may mean little by themselves, they are highly reproducible and highly repeatable. Results that are consistent from batch to batch give a good indication of product uniformity.

PENETRATION

(See "Grease Consistency")

PENTANE AND TOLUENE INSOLUBLES

When a used oil is diluted sufficiently with pentane, certain oxidation resins that it normally holds in solution are precipitated out. In addition, the dilution helps to settle out materials suspended in the oil. Among the latter are insoluble oxidation resins and extraneous matter such as dirt, soot, and wear metals. All of the contaminants that can be separated from the oil by precipitation and settling are referred to as *pentane insolubles*.

The pentane insolubles may then be treated with a toluene solution which dissolves the oxidation resins. The extraneous matter left behind is called the *toluene insolubles*. The difference between the pentane insolubles and the toluene insolubles represents the quantity of oxidation resins in the oil. This is termed the *insoluble resins*, meaning insoluble in pentane.

Toluene has replaced benzene as the aromatic solvent in ASTM D 893 because of concern about the potential toxicity of benzene. Insoluble sludges are generally similar with the two solvents.

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With detergent engine oils, a pentane-coagulant solution is customarily used instead of pentane. This precipitates material held in suspension by the detergent-dispersant which would not otherwise separate out.

As with other tests, interpretation depends on the type of oil, the service to which it has been put, and the results of other tests on the oil. In general, however, low pentane insolubles indicate an oil in good condition and little is to be gained by continuing with other phases of the test.

High pentane insolubles, on the other hand, indicate oxidation or contamination. The point at which an oil change is called for depends on many factors which must be evaluated by experience.

A relatively high value for toluene insolubles indicates contamination from an outside source such as soot from partially burned fuel; atmospheric dirt, the result of inadequate air filtration; tiny particles of metal produced by extreme wear. Emission spectrometry is often used to reveal the makeup of metal contamination: excessive lead, copper, or silver implies bearing wear; aluminum, piston wear; silicone, atmospheric dirt.

High insoluble resins mean a highly oxidized oil, which may result from excessive engine temperatures, contamination, an unsuitable oil, or excessive crankcase dilution. Loose-fitting piston rings, faulty injection, or low-temperature operation may allow raw fuel to enter the crankcase, where its oxidation adds to the amount of insoluble resins.

POUR POINT AND CLOUD POINT

ASTM D 97

It is often necessary to know how cold a particular petroleum oil can become before it loses its fluid characteristics. This information may be of considerable importance, for wide variations exist in this respect between different oils—even between oils of comparable viscosity.

If a lubricating oil is chilled sufficiently, it eventually reaches a temperature at which it will no longer flow under the influence of gravity. This condition may be brought about either by the thickening of the oil that always accompanies a reduction in temperature, or by the crystallization of waxy materials that are contained in the oil and that restrain the flow of the fluid portions. For many applications, an oil that does not flow of its own accord at low temperatures will not provide satisfactory lubrication. The extent to which an oil can be safely chilled is indicated by its *pour point*, the lowest temperature at which the undisturbed oil can be poured from a container.

The behavior of an oil at low temperature depends upon the type of crude from which it is refined, the method of refining, and the presence of additives. Paraffinic base stocks contain waxy components that remain completely in solution at ordinary temperatures. When the temperature drops, however, these waxy components start to crystallize, and they become fully crystallized at a temperature slightly below the pour point. At this temperature, the undisturbed oil will not generally flow under the influence of gravity.

Crystallization of the waxy component does not mean that the oil is actually solidified; flow is prevented by the crystalline structure. If this structure is ruptured by agita-

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tion, the oil will proceed to flow, even though its temperature remains somewhat below the pour point.

An oil that is predominantly naphthenic, on the other hand, reacts in a somewhat different manner. In addition to having a comparatively low wax content, a naphthenic oil thickens more than a paraffinic oil of comparable viscosity does when it is cooled. For these reasons, its pour point may be determined by the actual congealing of the entire body of oil instead of by the formation of waxy crystals. In such a case, agitation has little effect upon fluidity, unless it raises the temperature.

The pour point of a paraffinic oil may be lowered substantially by a refining process that removes the waxy component. For many lubricating oils, however, these components impart advantages in viscosity index and oxidation stability. Good performance generally establishes a limit beyond which the removal of these waxy component is inadvisable. It is possible, nevertheless, to lower the pour point of a paraffinic oil by the introduction of a pour depressant. Such an additive appears to stunt the growth of the individual crystals so that they offer less restriction to the fluid portions of the oil. It is hardly necessary to point out, however, that a pour depressant, as such, can have little, if any, effect upon a naphthenic oil.

Cloud point is the temperature, somewhat above the pour point, at which wax crystal formation gives the oil a cloudy appearance. Not all oils exhibit a cloud point and, although this property is related to pour point, it has little significance for lubricating oils. It is significant, however, for distillate fuels, and it is measured by ASTM D 2500.

Significance

The pour point of an oil is related to its ability to start lubricating when a cold machine is put in operation. Agitation by the pump will rupture any crystalline structure that may have formed, if the oil is not actually congealed, thereby restoring fluidity. But oil is usually supplied to the pump by gravity, and it can not be expected to reach the pump under these conditions, if the temperature is below the pour point. Passenger car engines and many machines that are stopped and started under low-temperature conditions require an oil that will flow readily when cold.

What is true of circulating lubrication systems, moreover, is equally true of gravity-feed oilers and hydraulic systems. A low pour point oil helps to provide full lubrication when the equipment is started and is easier to handle in cold weather. Low pour point is especially desirable in a transformer oil, which must circulate under all temperature conditions. The control of large aircraft is dependent upon hydraulic oils that must remain id after being exposed to extreme temperature drops. For these and similar applications, pour point is a very important consideration.

If the temperature of an oil does not drop below its pour point, the oil can be expected to flow without difficulty. It sometimes happens, however, that oil is stored for long periods at temperatures below the pour point. In some cases, the waxy crystalline structure that may be formed under these circumstances will not melt and redissolve when the temperature of the oil is raised back to the pour point. To pour the oil under these conditions, it is necessary to put the waxy crystals back in solution by heating the ell above its pour point.

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POWER FACTOR

ASTM D 924

Petroleum oils serve extensively as dielectrics for electrical power transmission equipment. Their primary functions are the cooling of coils (by circulation) and the prevention of arcing between conductors of high potential difference. In serving these purposes, any dielectric tends to introduce a degree of *dielectric loss*, a form of leakage equivalent to a flow of current through the dielectric from one conductor (wire or cable) to another. It is a leakage peculiar to a-c circuits. Though the loss associated with insulating oils is ordinarily a minor consideration, it could, under unusual conditions, assume a significant magnitude. In such a case, it would not only reduce the efficiency of the unit, but could cause a harmful rise in the unit's temperature.

Dielectric loss depends, among other things, on the nature and magnitude of the insulation's *impedance*, its opposition to the flow of alternating current through it. This is the current that is related to dielectric loss, and it increases as the impedance decreases. Only a portion of this current, however, is directly involved: a component equivalent to an *active* current. In a given a-c circuit, the loss is directly proportional to this active current. The ratio of active to *total* alternating current may vary—theoretically—from one to zero. This ratio is known as the *power factor* of the dielectric, and it can be considered to be an inherent dielectric property. Because of its effect on dielectric loss, the power factor of the dielectric should be as low as possible.

Though the power factor of an insulating oil is defined by the same mathematical expression as that of an a-c circuit, the two concepts should not be confused The overall power factor of a power-circuit affect *line* losses, rather than local dielectric losses, and the reduction of line losses requires a *high* power factor for the circuit.

Significance of Test Results

In a-c transmission cables, conductors of opposite polarity may extend for long distances in close proximity to each other. There is abundant opportunity for dielectric loss associated with the insulating material between the conductors. The higher the power factor of the insulation, the greater this loss will be.

For other applications, as in the insulation of transformers, dielectric loss is not appreciable, and small differences in power factor have little significance. A high-quality oil that is free of contamination can be expected to exhibit the low power factor that good performance requires.

In the evaluation of a used insulating oil's condition, however, power factor may be more meaningful. Here, the principal criterion is freedom from water and oxidation products—water that promotes the tendency to arc and oxidation sludges that interfere with cooling. Oxidation of the oil and contamination with water, dirt, or carbonized particles cause the power factor to rise.

Many engineers consider power factor to be a highly sensitive index of the oil's deterioration. If sufficient data on the performance of a particular oil in a particular service is available, it is possible to relate increases in power factor to degradation of the oil. In this way, power factor tests on a used oil may be helpful in estimating its remaining

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service life.

Tests for power factor frequently serve a useful purpose in the refinery as a check on uniformity of product quality. Consistent test values are indicative of consistent performance characteristics.

REFRACTIVE INDEX

ASM D 1218

Uniformity of composition of highly refined petroleum products is of importance, especially in process applications such as those involving solvents or rubber process oils. Refractive index is one test often used either alone or in combination with other physical tests as an indication of uniformity.

Refractive index is the ratio of the velocity of light of a specified wavelength in air to its velocity in a substance under examination. When light is passed through different petroleum liquids, for example, the velocity of light will be different in each liquid. Several sources of light of constant wavelength are available, but the yellow D line of sodium (5893 Å) is the one most commonly used in this test. Since the numerical value of the refractive index of a liquid varies with wavelength and temperature, it must be reported along with the wavelength and temperature at which the test was run.

This test is intended for transparent and light-colored hydrocarbon liquids having refractive indices between 1.33 and 1.50. The method is capable of measuring refractive index with a reproducibility of $\pm$ 0.00006. It is generally not this accurate with liquids having ASTM colors darker than 4 (ASTM D 1500), or with liquids that are so volatile at the test temperature that a reading cannot be obtained before evaporation starts.

Significance of Results

The refractive index is easily measured and possesses good repeatability and reproducibility. It is sensitive to composition. This makes it an excellent spot test for uniformity of composition of solvents, rubber process oils, and other petroleum products. A general rule for petroleum products of equivalent molecular weight is that paraffins have relatively low refractive indices (approximately 1.37), aromatics have relatively high indices approximately 1.50), and naphthalenes have intermediate indices (approximately 1.44).

Refractive index may be used in combination with other simple tests to estimate the distribution of carbon atom types in a process oil. Empirical refractive index charts relating viscosity, specific gravity, and refractive index have been prepared, and they make it possible to estimate the percent naphthenic, aromatic, and paraffinic carbon atoms sent. This is an inexpensive and quick set of tests to run, in contrast to the more time-consuming clay/silica gel analysis, which is also used to determine hydrocarbon comport directly.

ROTARY BOMB OXIDATION TEST (RBOT)

ASTM D 2272

Oxidation is a form of chemical deterioration to which petroleum products are sub-

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ject. Even though oxidation takes place at moderate temperatures, the reaction accelerates significantly at temperatures above 200°F.

In addition to the effect of high temperatures, oxidation may also be speeded by catalysts (such as copper) and the presence of water, acids, or, solid contaminants. Moreover, the peroxides that are the initial products of oxidation are themselves oxidizing agents, so that oxidation is a chain-reaction—the further it progresses the more rapid it becomes.

Even though subject to oxidation, many oils (such as turbine oils) give years of service without need for replacement. Petroleum products can be formulated to meet service and storage life requirements by: (1) proper selection of crude oil type; (2) thorough refining, which removes the more-oxidation-susceptible materials; and (3) addition of oxidation inhibitors.

A number of oxidation tests are currently being used. Some may be better related to a particular type of service than others. All are intended to simulate service conditions on an accelerated basis. The most familiar method is the "Oxidation Characteristics of Inhibited Steam-Turbine Oils," ASTM D 943. The long time (over 1000 hours) required to run this test makes it impractical for plant control work. ASTM Method D 2272, "Continuity of Steam-Turbine Oil Oxidation Stability by Rotating Bomb," on the other hand, allows rapid evaluation of the resistance of lubricants to oxidation and sludge formation, using accelerated test conditions that involve high temperatures, high-pressure oxygen, and the presence of water and catalytically active metals.

The "rotary bomb oxidation test" (RBOT) does not replace ASTM D 943, but is intended primarily as an aid in quality control during the manufacture of long-life circulating oils.

Significance of Results

The ASTM D 2272 procedure allows relative oxidation life of a turbine oil to be determined rapidly. Results are obtained by the RBOT test up to 1000 times faster than by the D 943 method. This speed makes the test practical for use as a product quality control measure, permitting decisions to be made within a matter of a few hours. The RBOT is also distinguished among oxidation tests by its good repeatability and reproducibility.

It should be remembered that the test is essentially a quality control device and no direct correlation has been established with other oxidation tests currently being used. For two oils of similar composition—both base stock and additive package—the RBOT test can be used as an indication of their relative oxidation stability.

RUST-PREVENTIVE CHARACTERISTICS

ASTM D 665

This test method was originally designed to indicate the ability of steam-turbine oils to prevent the rusting of ferrous parts, should water become mixed with the oil. While still used for this purpose, its application is now often extended to serve as an indication of rust preventive properties of other types of oils, particularly those used in circulating systems.

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It is a dynamic test, designed to simulate most of the conditions of actual operation.

In the method, a standard steel specimen is immersed in a mixture of the test oil and water under standard conditions and with constant stirring. At the end of a specified period, the steel specimen is examined for rust. Depending on the appearance of the specimen, the oil is rated as passing or failing.

Degrees of Rusting

An indication of the degree of rusting occurring in this test is sometimes desired. For such cases, the following classification is recommended:

LIGHT RUSTING—Rusting confined to not more than six spots, each of which is 1 mm or less in diameter.

MODERATE RUSTING—Rusting in excess of the preceding, but confined to less than 5% of the surface of the specimen.

SEVERE RUSTING—Rust covering more than 5% of the surface of the specimen.

Reporting Results

Results obtained with a given oil are reported as "pass" or "fail." Since the test may be conducted with either distilled water or with synthetic sea water, and for varying periods of time, reports of results should always specify these conditions. For example: "Rust Test, ASTM D 665, Procedure B, 24 Hours—Pass."

Significance

When the lubricating oil of a turbine or other system is contaminated with water, rusting can result. Particles of rust in the oil can act as catalysts that tend to increase the rate of oil oxidation. Rust particles are abrasive, and cause wear and scoring of critical parts. In addition, rust particles can add to other contaminants in a circulating system, increasing the tendency toward the clogging of low-clearance members, such as servo valves, and increasing the probability of filter plugging.

In many cases, the rusting characteristics of the system in service are better than is indicated by testing a sample of the used oil, because the polar rust inhibitor "plates out" on the metal surfaces (which are therefore adequately protected). The sample of oil, being somewhat depleted of the inhibitor, will then allow greater rusting in the test than would occur in service.

The relative ability of an oil to prevent rusting can become a critical property in many applications. As noted, this test method was originally applied exclusively to steam turbine oils. However, the test is now frequently applied to other oils in different types of applications, whenever undesirable water contamination is a possibility.

SAPONIFICATION NUMBER

Many lubricating oils are "compounded" with fatty materials to increase their film strengths or water-displacing qualities. The degree of compounding is indicated by the

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saponification number of the oil, usually called its *sap number*. Sap number is commonly determined by ASTM D 94 or D 939, methods based on the fact that these fatty materials can be saponified—that is, converted to soap—by reaction with a base (alkali), usually at an elevated temperature.

A specified quantity of potassium hydroxide (KOH) is added to the prepared oil sample and the mixture heated to bring about reaction. The excess KOH is titrated to neutralization with hydrochloric acid, either colorimetrically (D 94) or potentiometrically (D 939). The sap number is reported as milligrams of KOH assimilated per gram of oil.

Other factors being the same, a higher degree of compounding will result in a higher sap number. For a given degree of compounding, however, some fatty materials show a higher sap number than others.

Even with a new oil, therefore, sap number cannot be translated directly into percentage of fatty materials unless their exact nature is known.

Considerable experience with a particular set of conditions and types of oil is needed to properly interpret the sap number of a used oil. While loss or decomposition of fatty materials is reflected in a drop of the sap number, oxidation of the mineral oil base may cause the sap number to rise. Test results may be further distorted by acid or metallic contaminants picked up in service.

It is advisable, therefore, to consider sap number in relation to neut number. Comparison of the two indicates what portion of the sap number is due to the presence of fatty materials and what portion to acids in the oil.

Sap number has little relevance for oils for internal combustion engines.

TIMKEN EXTREME PRESSURE TESTS

ASTM D 2509—Lubricating Greases
ASTM D 2782—Lubricating Fluids

(See "Load Carrying Ability.")

USP/NF TESTS FOR WHITE MINERAL OILS

The *US Pharmacopeia* and the *Natioiwl Formulary*, publications by two independent associations of physicians and pharmacologists, contain specifications for white mineral oils. The US Pharmacopeia (USP) specification covers the more viscous "Mineral Oil," which is used primarily as a pharmaceutical aid or levigating agent. The National Formulary (NF) sets specifications for the less viscous "Light Mineral Oil," which is used as a vehicle in drug formulations. Both compendia have legal status, being recognized in federal statutes, especially the Federal Food, Drug and Cosmetic Act.

White mineral oils have certain physical properties that distinguish them from other petroleum products. Both the USP and NF describe them as colorless, transparent, oily liquids free or nearly free from fluorescence. When cold they are odorless and tasteless and develop only a slight petroleum odor when heated. They are insoluble in water and alcohol, soluble in volatile oils, and miscible with most fixed oils with the exception of castor oil.

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Significance of Results

These tests are designed to establish standards that assure that the oils involved are pure, chemically inert, and free from potentially carcinogenic materials. Oils that meet these standards find use, not only in pharmaceuticals and cosmetics, but also in chemical, plastics, and packaging applications where they are considered as direct or indirect food additives as defined by the Federal Food and Drug Administration (FDA).

UV ABSORBANCE

FDA Method

Petroleum product applications often extend into areas other than the obvious ones. One such area is the direct or indirect application of a petroleum product to food. A direct food additive is one that is incorporated, in small quantities, into or onto food meant for human or animal consumption. An example would be the use of white mineral oil to coat raw fruits to protect them or to coat animal feeds to reduce dustiness. An indirect additive is one that has only incidental contact with food, as through contact with a packaging material.

The use of petroleum products as food additives falls under the jurisdiction of the Food and Drug Administration. A major concern in the regulation of food additives of petroleum origin is the potential contamination of food by polynuclear aromatic hydrocarbons—some of which are considered to be carcinogenic. In an attempt to assure the absence of carcinogens, the FDA has sanctioned the use of ultraviolet (UV) absorbance as a test for monitoring the polynuclear aromatics content.

Ultraviolet absorbance is a measure of the relative amount of ultraviolet light absorbed by a substance. Types of compounds can be characterized by the wavelength range of UV light that they absorb. As the wavelength of ultraviolet light is varied, broad peaks of absorbance occur at the wavelengths that are characteristic of the compounds present. Most polynuclear aromatics have their principal absorbances at wavelengths between 280 and 400 millimicrons. Most carcinogens absorb UV light in this range, but not all materials with UV absorbance between 280 and 400 millimicrons are carcinogenic.

Significance of Results

These results are compared with the corresponding UV absorbance limits set by the FDA for the specific regulation that applies to each UV. When UV absorbance of a petroleum product falls within these limits, the product is considered acceptable for the particular food application involved. The UV method described here represents the simplest case. The method becomes more complex as the aromatic concentration of the oil increases.

The UV test is not the only criterion the FDA has established for food additives of petroleum origin. There are often additional requirements for boiling range, color, odor, method of manufacture, US Pharmacopeia quality, and non-volatile residues.

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VAPOR PRESSURE

ASTM D 323

All liquids are disposed to vaporize—that is, to become gases. This tendency is a manifestation of the material's liquid vapor pressure, the pressure exerted by molecules at the liquid surface in their attempt to escape and to penetrate their environment. For a given liquid, this pressure is a function purely of temperature. The liquid vapor pressure of water at is boiling temperature—212°F—for example, is 147 psi, the pressure of the atmosphere. The more volatile the liquid, the higher the liquid vapor pressure at a specified temperature, and the faster the vaporization. In the same dry atmosphere and at the same liquid temperature, gasoline evaporates much more readily than heating oil.

For a given temperature, therefore, the vapor pressure of a liquid is a measure of is volatility. This applies only to vapor pressure exerted by a *liquid*. Pressures exerted by vapor disassociated from the liquid are functions of *volume*, as well as temperature, and they cover a wide range of values less directly related to volatility. As used in engineering circles, the term vapor pressure means *liquid* vapor pressure.

Unlike water, a petroleum product usually comprises many different fractions, each with a composition and a vapor pressure of its own. The vapor pressure of the product is therefore a composite value that reflects the combined effect of the individual vapor pressures of the different fractions in accordance with their mole ratios. It is thus possible for two wholly different products to exhibit the same vapor pressure at the same temperature—provided the cumulative pressures exerted by the fractions are also the same. A narrow-cut distillate, for example, may exhibit the same vapor pressure as that of a dumbbell blend, where the effect of the heavy fractions is counterbalanced by that of the lighter ones.

When a petroleum product evaporates, the tendency is for the more volatile fractions to be released first, leaving a material of lower vapor pressure and lower volatility behind. This accounts for the progressive rise in distillation-curve temperature, boiling point being related to volatility. Distillation, which is another measure of volatility, was described earlier.

Vapor pressure is commonly measured in accordance with the ASTM method D 323 (Reid vapor pressure), which evaluates the vapor pressures of gasoline and other volatile petroleum products at 100°F.

Significance of Test Results

Reid vapor pressure has a special significance for gasoline, which contains a portion of high-volatility fractions such as butane, pentane, etc. These fractions exert a major influence on vapor-pressure test results. A high vapor pressure is accordingly an indication of the presence of these high-volatility fractions—components required for satisfactory starting in cold weather. Without them, it would be difficult or impossible to vaporize gasoline in sufficient concentration to produce a combustible air-fuel mixture at low temperatures.

On the other hand, vapor pressure may be too high. An excess of high-volatility actions in hot weather can lead to vapor *lock*, preventing delivery of fuel to the carbure-

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tor. This is the result of the partial vacuum that exists at the suction end of the fuel pump and that, along with high temperatures, increases the tendency of the fuel to vaporize. If the fuel vapor pressure is too high, vapors formed in the suction line will interrupt the flow of liquid fuel to the pump, causing the engine to stall.

While Reid vapor pressure is the principal factor in determining both the vapor-lock and the cold-starting characteristics of a gasoline, they are not the only criteria. Distillation data, which defines the overall volatility of the fuel, must also be considered.

The higher the vapor pressures of automotive and aviation gasolines, solvents, and other volatile petroleum products, the greater the possibility of evaporation loss and the greater the fire hazard. Sealed containers for high-vapor-pressure products require stronger construction to withstand the high internal pressure. In the refinery, moreover, vapor pressure tests serve as a means of establishing and maintaining gasoline quality.

VISCOSITY

ASTM D 88, D 445, Redwood, and Engler

Viscosity is probably the most significant physical property of a petroleum lubricating oil. It is the measure of the oil's flow characteristics. The thicker the oil, the higher its viscosity, and the greater its resistance to flow. The mechanics of establishing a proper lubricating film depend largely upon viscosity.

To evaluate the viscosity of an oil numerically, any of several standard tests may be applied. Though these tests differ to a greater or lesser extent in detail, they are essentially the same in principle. They all measure the time required for a specified quantity of oil at a specified temperature to flow by gravity through an orifice or constriction of specified dimensions. The thicker the oil, the longer the time required for its passage.

Close control of oil temperature is important. The viscosity of any petroleum oil increases when the oil is cooled and diminishes when it is heated. For this same reason, the viscosity value of an oil must always be accompanied by the temperature at which the viscosity was determined. The viscosity value by itself is meaningless.

The two most common methods of testing the viscosity of a lubricating oil are the Saybolt and the kinematic. Of these, the Saybolt (ASTM D 88) is the method more frequently encountered in conjunction with lubricating oils. However, the kinematic method (ASTM D 445) is generally considered to be more precise. There are also the Redwood and the Engler methods, which are widely used in Europe, but only to a limited extent in the United States. Each test method requires its own apparatus—viscosimeter (or viscometer).

Significance of Results

Viscosity is often the first consideration in the selection of a lubricating oil. For most effective lubrication, viscosity must conform to the speed, load, and temperature conditions of the bearing or other lubricated part. Higher speeds, lower pressures, or lower temperatures require an oil of a lower viscosity grade. An oil that is heavier than necessary introduces excessive fluid friction and creates unnecessary drag.

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Lower speeds, higher pressures, or higher temperatures, on the other hand, require an oil of a higher viscosity grade. An oil that is too light lacks the film strength necessary to carry the load and to give adequate protection to the wearing surfaces. For these reasons, viscosity tests play a major role in determining the lubricating properties of an oil.

In addition to the direct and more obvious conclusions to be drawn from the viscosity rating of an oil, however, certain information of an indirect sort is also available. Since, to begin with, the viscosity of the lube oil cut is determined by its distillation temperature, it is apparent that viscosity and volatility are related properties. In a general way, the lighter the oil, the greater its volatility—the more susceptible it is to evaporation. Under high-temperature operating conditions, therefore, the volatility of an oil, as indicated by its viscosity, should be taken into consideration.

Though the significance of viscosity test results has been considered from the standpoint of new oils, these tests also have a place in the evaluation of used oils. Oils drained from crankcases, circulating systems, or gear boxes are often analyzed to determine their fitness for further service or to diagnose defects in machine performance.

An increase in viscosity during service may often indicate oxidation of the oil. Oxidation of the oil molecule increases its size, thereby thickening the oil. When oxidation has progressed to the point of causing a material rise in viscosity, appreciable deterioration has taken place.

VISCOSITY CLASSIFICATIONS COMPARISON

There are four common systems for classifying the viscosities of lubricating oils. It is frequently desirable to compare a grade in one system with a grade in another system, but this is often impossible because the standards in the different systems are not based on viscosities at the same temperature. The charts presented in Figures 3-2 and 3-3 are designed to overcome this problem by comparing the systems on the basis of viscosities at a single temperature—100°F, which is the base temperature for the ASTM viscosity grade system.

In order to convert all viscosities to 100°F, it is necessary to assume appropriate viscosity indices (VI's) for the oils involved. (Viscosity index of an oil is a measure of its resistance to change in viscosity as temperature changes.) The VI's assumed here are:

110 VI for crankcase oils (SAE)
90 VI for automotive gear oils (SAE)

These values are representative for the products involved in the respective classifications. Close comparison should not be attempted if the VI of the product differs appreciably from the values used.

Figure 3-2 shows the numerical relationships; Figure 3-3 shows the graphical equivalents.

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Figure 3-2. Numerical relationships among viscosity classification systems.

ASTM Industrial Oils	Viscosity, SSU at 100°F	SAE Crankcase Oils	Viscosity, SSU at 100°F (assumes 110 VI)	AGMA Industrial Gear Oils	Viscosity SSU at 100°F	SAE Gear Oils	Viscosity at 100°F (assumes 90 VI)
32	33-34						
36(A)	36-38						
40	40-43						
50(A)	46-50	5W	140 Max.			75	211 Max.
60	55-62						
75	72-83						
105	97-116						
150	136-165	10W	140-220				
215	193-235	20	168-366	1	193-234		
315	284-347	20W	202-500	2,2EP	284-347	80	211-575
465	417-510	30	366-560	3,3EP	417-510		
700	625-764	40	560-812	4,4EP	626-765		
1000	917-1121	50	812-1272	5,5EP	916-1122	90	768-1844
1500	1334-1631	60(B)	1272-1561	6,6EP	1335-1632		
2150	1918-2344	70(B)	1561-2085	7EP,7 Comp.	1919-2346		
						140	1844-4345
3150	2835-3465			8EP,8 Comp.	2837-3467		
4650	4169-5095			8A Comp.	4171-5098		
						250	4345 Min.
7000	6253-7643						

Notes: (A) Currently is not considered a standardized grade in the USA. (B) Not officially recognized by SAE.

***ASTM/BSI** (American Society for Testing and Materials/British Standards Institution) "Viscosity Classification **System** for Industrial Fluid Lubricants."

SAE (Society of Automotive Engineers) "Crankcase Oil Viscosity Classification" and "Transmission and **Axle** Lubricant Classification."

AGMA (American Gear Manufacturers Association) "Specification—Lubrication of Industrial Enclosed **Gear** Drives."

VISCOSITY INDEX

ASTM D 567 and D 2270

Liquids have a tendency to thin out when heated and to thicken when cooled. However, this response of viscosity to temperature changes is more pronounced in some liquids than in others.

Often, as with petroleum liquids, changes in viscosity can have marked effects upon the performance of a product, or upon its suitability for certain applications. The prop-

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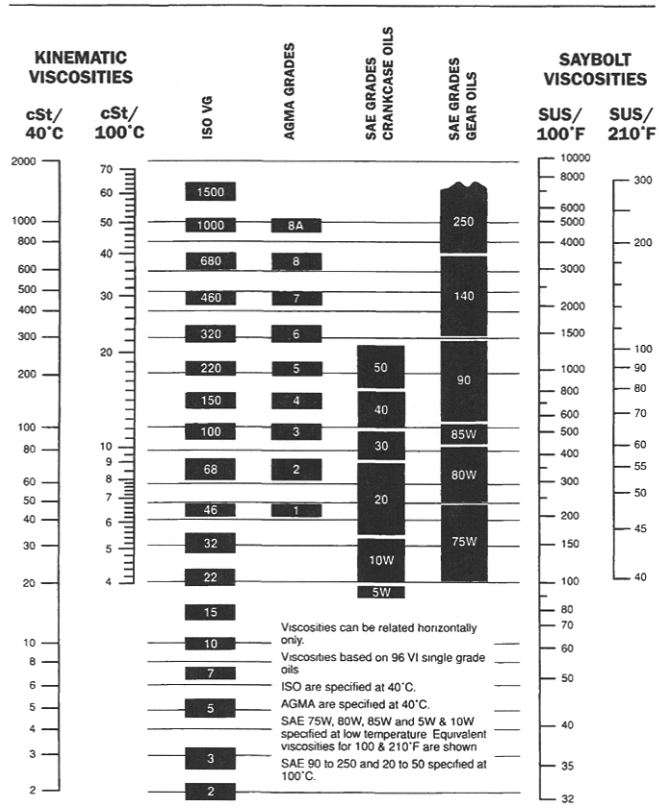

ISO VISCOSITY CLASSIFICATION SYSTEM

Many petroleum products are graded according to the ISO Viscosity Classificaiton System, approved by the International Standards Organization (ISO). Each ISO viscosity grade number corresponds to the mid-point of a viscosity range expressed in centistokes (cSt) at 40°C. For example, a lubricant with an ISO grade of 32 has a viscosity within the range of 28.8-35.2, the midpoint of which is 32.

Rule-of-Thumb: The comparable ISO grade of a given product whose viscosity in SUS at 100°F is known can be determined by using the following conversion formula:

SUS @ 100°F ÷ 5 ≅ cSt @ 40°C

Figure 3-3. Viscosity classification equivalents.

erty of resisting changes in viscosity due to changes in temperature can be expressed as the viscosity index (V.I.). The viscosity index is an empirical, unitless number. The higher the V.I. of an oil, the less its viscosity changes with changes in temperature.

The Concept of Viscosity Index

One of the things that led to the development of a viscosity index was the early observation that, for oils of equal viscosities at a given temperature, a naphthenic oil thinned out more at higher temperatures than did a paraffinic oil. However, there existed no single parameter that could express this type of response to temperature changes.

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The viscosity index system that was developed to do this was based upon comparison of the viscosity characteristics of an oil with those of so-called "standard" oils. A naphthenic oil in a series of grades with different viscosities at a given temperature, and whose viscosities changed a great deal with temperature, was arbitrarily assigned a V.I. of zero. A paraffinic series, whose viscosities changed less with temperature than most of the oils that were then available, was assigned a V.I. of 100. With accurate viscosity data on these two series of oils, the V.I. of any oil could be expressed as a percentage factor relating the viscosities at 100°F of the test oil, the zero-V.I. oil, and the 100-V.I. oil, all of which had the same viscosity at 210°F. This is illustrated by Figure 3-4 and is the basis for the formula,

$$\text{V.I.} = \frac{L-U}{L-H} \times 100$$

where L is the viscosity at 100°F of the zero-V.I. oil, H is the viscosity at 100°F of the 100-V.I. oil, and U is the viscosity at 100°F of the unknown (test) oil.

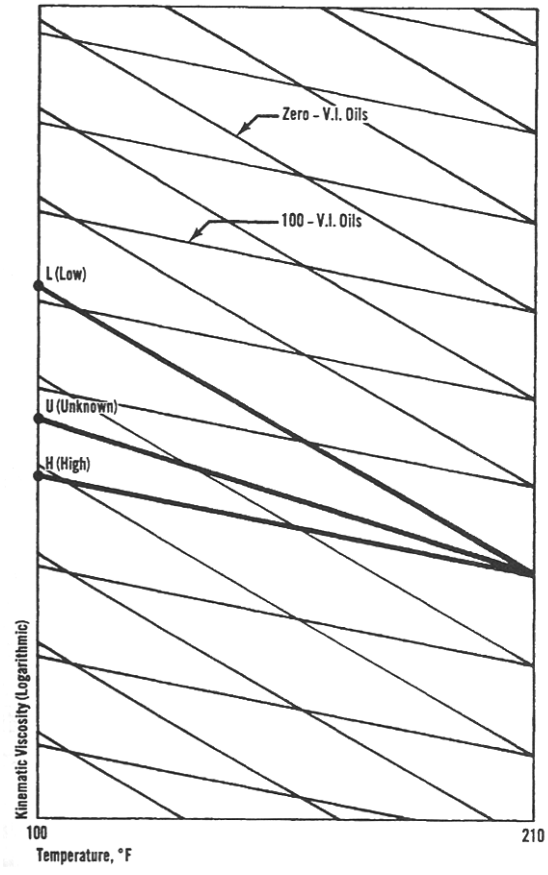

Figure 3-4. The concept of viscosity index.

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The ASTM Standards

This viscosity index system eventually became the ASTM standard D 567, which has been used for years in the petroleum industry.

ASTM D 567 is a satisfactory V.I. system for most petroleum products. However, for V.I.'s above about 125, mathematical inconsistencies arise which become more pronounced with higher V.I.'s. Because products with very high V.I.'s are becoming more common, a method (ASTM D 2270) that eliminates these inconsistencies has been developed.

Calculating Viscosity Index

The viscosity index of an oil can be calculated from tables or charts included in the ASTM methods. For V.I.'s below 100, ASTM D 2270 and ASTM D 567 are identical, and either method may be used. For V.I.'s above 100, ASTM D 2270 should be used. Since ASTM D 2270 is suitable for all V.I.'s, it is the method now preferred by the leading petroleum companies.

The V.I. of an oil may also be determined with reasonable accuracy by means of special nomographs or charts developed from ASTM tables. A chart for V.I.'s above 100, as determined by ASTM D 2270, is shown in Figure 3-5.

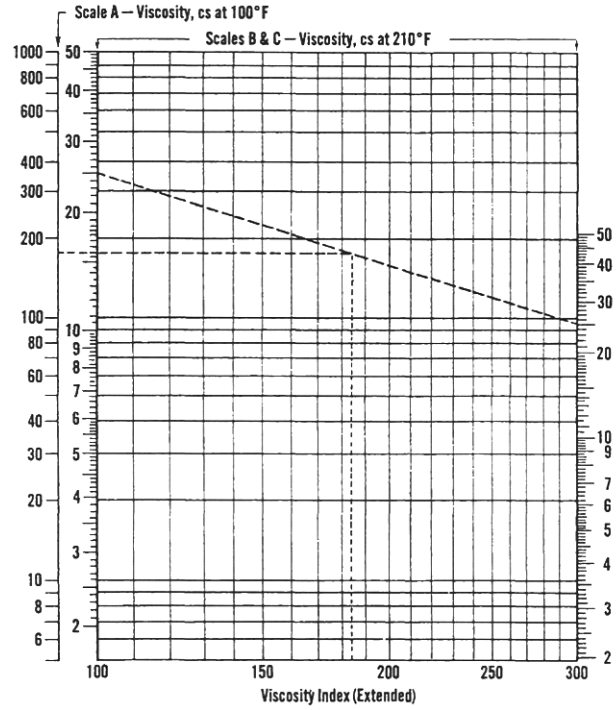

Figure 3-5. Chart for calculating V.I.'s above 100 from kinematic viscosity, based on ASTM D 2270. Dotted lines illustrate its use.

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Significance of Viscosity Index

Lubricating oils are subjected to wide ranges of temperatures in service. At high temperatures, the viscosity of an oil may drop to a point where the lubricating film is broken, resulting in metal-to-metal contact and severe wear. At the other extreme, the oil may become too viscous for proper circulation, or may set up such high viscous forces that proper operation of machinery is difficult. Consequently, many applications require an oil with a high-viscosity index.

In an automobile, for example, the crankcase oil must not be so thick at low starting temperatures as to impose excessive drag on the engine and to make cranking difficult. During the warm-up period, the oil must flow freely to provide full lubrication to all engine parts. After the oil has reached operating temperature, it must not thin out to the point where consumption is high or where the lubricating film can no longer carry its load.

Similarly, fluid in an aircraft hydraulic system may be exposed to temperatures of 100°F or more on the ground, and to temperatures well below zero at high altitudes. For proper operation under these varying conditions, the viscosity of the hydraulic fluid should remain relatively constant, which requires a high viscosity index.

As suggested by the relationship between naphthenic and paraifinic oils, the viscosity index of an oil can sometimes be taken as an indication of the type of base stock. A straight mineral oil with a high V.I.—80 or above—is probably paraffinic, while a V.I. below about 40 usually indicates a naphthenic base stock.

In general, however, this relationship between V.I. and type of base stock holds only for straight mineral oils. The refining techniques and the additives that are available today make it possible to produce naphthenic oils with many of the characteristics—including V.I.—of paraffinic oils. V.I., then, should be considered an indication of hydrocarbon composition only in the light of additional information.

WATER WASHOUT

ASIM D 1264

Lubricating greases are often used in applications that involve operation under wet conditions where water may enter the mechanism and mix with the grease. Therefore, the ability of a grease to resist washout becomes an important property in the maintenance of a satisfactory lubricating film, and tests for evaluating the effect of water on grease properties are of considerable interest.

Greases can be resistant to water in several ways. Some greases completely reject the admixture of water or may retain it only as occluded droplets with little change in structure. Unless these greases are adequately inhibited against rusting they may be unsuitable for lubrication under wet conditions since the "free" water could contact the metal surface and cause rusting.

Yet other greases that absorb water may be satisfactory under wet conditions. These types of grease absorb relatively large amounts of water by forming emulsions of water in oil. This absorption has little effect on the grease structure and leaves no "free"

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water to wet and rust the metal. Therefore, the grease continues to supply the proper lubrication while also acting as a rust preventive.

Other water-absorbing greases form thin fluid emulsions so that the grease structure is destroyed. These are useless for operation under wet conditions, and can be considered to have poor water resistance.

There are many effects that water has on grease, and no single test can cover them all. Many of the tests are useful tools; however, the results are subject to the personal judgment of the test operator and much skill is needed to interpret their meaning. ASTM Method D 1264, "Water Washout Characteristics of Lubricating Greases," is one method of evaluating this property.

Significance

Test results are useful for predicting the probable behavior of a grease in a shielded (not positively sealed) bearing exposed to the washing action of water. They are a measure of the solubility of a grease in water and give limited information on the effect of water on the grease structure. They say nothing about the rust preventive properties of the grease.

The test is a laboratory procedure and should not be considered equivalent to a service evaluation. Results on greases tested by this method may differ from service results because of differences in housing or seal design. Therefore a grease that proves unsatisfactory according to this test, may be satisfactory under service conditions if the housing or seal design is suitable.

WATER AND SEDIMENT

ASTM D 96, D 95, and D 473

Whether a petroleum fuel is burned in a boiler or in an engine, foreign matter in the fuel is undesirable. Excessive quantities of such impurities as water or solid contamination may interrupt the operation of the unit, and may also damage it.

The two most common impurities found in fuel oils are water and sediment, and several test procedures are available for measuring their concentrations. Water and sediment may be determined together by a centrifuge procedure. Water alone may be determined more accurately in most cases by distillation, and sediment alone may be determined with good accuracy by solvent extraction or by hot filtration.

The tests referred to are the following:

(1) ASTM D 96 Water and Sediment in Crude Oils
(2) ASTM D 95 Water in Petroleum Products and Other Bituminous Materials
(3) ASTM D 473 Sediment in Crude Petroleum Fuel Oil by Extraction

Significance of Results

Like many other tests, the determination of water and sediment gives results that must be interpreted in the light of a great deal of previous experience. It is obvious that large quantities of water and/or sediment can cause trouble in almost any application.

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However, different applications can tolerate different concentrations of impurities. In addition, the quantities of water and sediment determined by the various test procedures are not identical.

Therefore, for any particular application, it is necessary to determine the relation between the tolerance of that application and the results of one or more tests. When this has been done, these tests may be used as controls for that application.

It should be remembered that although a petroleum product may be clean when it leaves the refinery, it is possible for it to pick up contamination from the storage and handling equipment and practices, or as a result of condensation. Water and sediment are often picked up in tanks of ships and in other types of transportation or storage facilities.

WAX MELTING POINT

Melting Point (Plateau) of Petroleum Wax (ASTM D 87)
Drop Melting Point of Petroleum Wax (ASTM D 127)
Congealing Point of Petroleum Wax (ASTM D 938)

Each of the three test methods discussed here provide information about the transition between the solid and liquid states of petroleum waxes. The tests differ, however, by procedure and in the types of material to which they are applicable.

Both ASTM D 87 and ASTM D 127 are designed to determine the temperature at which most of the wax sample makes the transition between the liquid and the solid states. ASTM D 87 is applicable only to materials that show a "plateau" on their cooling curve. This plateau occurs when the temperature of a material passing into the solid phase remains constant for the time required to give up heat of fusion.

ASTM D 127 determines the temperature at which the material becomes sufficiently fluid to drip. The melting points of high-viscosity waxes that do not show a plateau can be determined by this method.

As determined by ASTM D 938, the congealing point is the temperature at which molten wax ceases to flow.

Significance

All three of the test methods are found in common specifications and buying guides among industries using large volumes of wax. The choice of a particular test method depends on the nature of the wax and the application.

Petroleum waxes are mixtures of hydrocarbon materials having different molecular weights. If these materials crystallize at about the same temperature, the cooling curve of the wax will show a plateau. ASTM D 87 is applicable to such waxes. Microcrystalline waxes, however, do not show a plateau in their cooling curve. The melting point of these waxes is usually reported by ASTM D 127. In general, ASTM D 127 is best suited for high-viscosity petroleum waxes.

The congealing point of a wax is usually slightly lower than either of its melting points. Congealing point (ASTM D 938) is often used when storage or application temperature is a critical factor, since it will give a more conservative estimate of the level at which temperatures should be maintained.

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WHEEL BEARING GREASE LEAKAGE

ASTM D 1263

Under actual service conditions automotive front wheel bearings frequently operate at high temperatures. This is caused by a combination of heavy loads, high speeds, and the heat generated by braking. Because of this, greases used to lubricate these wheel bearings must be resistant to softening and leaking from the bearing. ASTM Method D 1263, "Leakage Tendencies of Automotive Wheel Bearing Greases," is an evaluation of leakage tendencies under prescribed laboratory test conditions.

Apparatus

The test apparatus consists of a special front wheel hub and spindle assembly encased in a thermostatically controlled air bath. Grease that leaks from the bearings is collected in the hub cap and leakage collector. Means for measuring both ambient and spindle temperatures are provided.

Significance

This test is an accelerated leakage test and is mainly a measure of the ability of a grease to be retained in the bearings at the test temperature. However, experienced operators can observe other changes in grease condition such as softening or slumping, but these are subjective judgments and not readily expressed in quantitative terms. There is no load or vibration applied to the bearings such as exists in normal wheel bearing service, and the test temperature of 220°F may be considerably lower than encountered in modern vehicles equipped with disc brakes. These factors are recognized and other test devices and procedures are under study by ASTM. The test is of primary value as a screening procedure to be used in conjunction with other stability tests in the development and evaluation of new grease formulations. Because of its limited sensitivity and precision, it permits differentiation only among products of distinctly different leakage characteristics.

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